PB85-117034

Mobility of Toxic Compounds from Hazardous Wastes

Oak Ridge National Lab., TN

Prepared for

Environmental Monitoring Systems Lab.
Las Vegas, NV

Jul 84

U.S. Department of Commerce
National Technical Information Service
NTIS

TECHNICAL REPORT DATA

(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/4-84-069

2.

3. RECIPIENT'S ACCESSION NO. PB85 117034

4. TITLE AND SUBTITLE MOBILITY OF TOXIC COMPOUNDS FROM HAZARDOUS WASTES

5. REPORT DATE August 1984

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S) C. W. Francis, M. P. Maskarinec, and J. C. Goyert

8. PERFORMING ORGANIZATION REPORT NO.

9. PERFORMING ORGANIZATION NAME AND ADDRESS

Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

10. PROGRAM ELEMENT NO. ABSD1A

11. CONTRACT/GRANT NO. IAG's DOE No. 40-1087-80 and DW 8993007-01-2

12. SPONSORING AGENCY NAME AND ADDRESS

U.S. Environmental Protection Agency--Las Vegas, NV
Office of Research and Development
Environmental Monitoring Systems Laboratory
Las Vegas, Nevada 89114

13. TYPE OF REPORT AND PERIOD COVERED Project, 1/82 - 12/83

14. SPONSORING AGENCY CODE EPA/600/07

15. SUPPLEMENTARY NOTES

16. ABSTRACT

The objective of this research was to develop a laboratory extraction method for solid wastes that simulates concentrations of inorganic and organic constituents in leachates that result from co-disposing industrial wastes with municipal wastes in landfills (co-disposal in a landfill containing a 95:5 ratio of municipal to industrial waste, respectively). The intent was to produce a scientific rationale and a data base from which an extraction method can be selected to simulate leaching of solid wastes co-disposed with municipal wastes.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group

18. DISTRIBUTION STATEMENT RELEASE TO PUBLIC

19. SECURITY CLASS *(This Report)* UNCLASSIFIED

20. SECURITY CLASS *(This page)* UNCLASSIFIED

21. NO. OF PAGES 286

22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

PB85-117034

EPA-600/4-84-069
July 1984

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Interagency Agreements
Between
U.S. Department of Energy
DOE No. 40-1087-80
U.S. Environmental Protection Agency
DW 8993007-01-2

Project Officer

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Quality Assurance Division
Environmental Monitoring Systems Laboratory
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ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
LAS VEGAS, NEVADA 89114

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NATIONAL TECHNICAL
INFORMATION SERVICE
U.S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

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ACRONYMS

AAS	Atomic Absorption Spectroscopy
AMC	Average Maximum Concentration
API	American Petroleum Institute
ASTM	American Society of Testing Materials
CFR	Code of Federal Regulations
COD	Chemical Oxygen Demand
EP	Extraction Procedure
EPA	Environmental Protection Agency
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HCB	Hexachlorobutadiene
HPLC	High-Pressure Liquid Chromatography
IAC	Integrated Average Concentration
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
MLC	Maximum Leachate Concentration
MWL	Municipal Waste Leachate
NAA	Neutron Activation Analysis
NBS	National Bureau of Standards
NIPDWS	National Interim Primary Drinking Water Standard
ODS	Octadecylsilane
ORNL	Oak Ridge National Laboratory
PAH	Polynuclear Aromatic Hydrocarbons
PTFE	Polytetraflouroethylene
RCRA	Resource Conservation and Recovery Act
TOC	Total Organic Carbon
USGS	United States Geological Survey
UV	Ultraviolet
WES	Waterways Experimental Station

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INTRODUCTION

BACKGROUND

To evaluate the potential threat to ground water posed by improper disposal of an industrial waste in a sanitary landfill with municipal waste, a laboratory extraction procedure is needed that models the concentrations of organic and inorganic constituents in the leachate of that waste. Currently, under the Resource Conservation and Recovery Act (RCRA), such mobility is determined by an extraction procedure (EP) toxicity test (USEPA 1980a).

The EP is a 24-h batch-type laboratory extraction procedure that uses acetic acid to acidify the liquid/solid suspension (20:1 ratio) to a pH of 5 (maximum concentration of acetc acid is 2 meq/g of waste). The intent of the EP is to simulate the leaching action of acetic acid, the dominant carboxylic acid found in municipal waste leachate (MWL). A complete description of the required equipment and procedures to conduct an EP is described in USEPA (1982).

Hazardous waste identification regulatory thresholds for the EP toxicity characteristics (USEPA 1980a) are based on the concentrations of eight elements (As, Ba, Cd, Cr, Pb, Hg, Se, and Ag), four pesticides (Endrin, Lindane, Methoxychlor, and Toxaphene), and two herbicides (2,4-D and 2,4,5-TP Silvex) for which National Interim Primary Drinking Water Standards (NIPDWS) have been established.

The EP, a regulatory test used to identify hazardous wastes based on a landfill management scenario, has a number of limitations. The most important are (1) its ability to simulate a real-world disposal environment has not been tested and (2) solid waste leachates containing toxic constituents other than those listed above are not included in the current criteria. Also, it is uncertain whether the EP models the leaching of both polar and nonpolar organic compounds that would result with an extractant containing higher molcular weight acids than acetic acid. Other experimental factors that may limit the ability of the EP to accurately characterize potential health and environmental hazards of a waste include the deficiency in expressing kinetic relationships of components extracted, the relevance to the leaching in anoxic environments, and its limited applicability to bioassays. In terms of applying biological testing to EP extracts, the EP is limited because the acetic acid used in the procedure has been shown to interfere with certain aquatic toxicity and phytotoxicity testing protocols (Epler et al. 1980, Millemann et al. 1981).

OBJECTIVES

The research objective is to develop an experimental data base from which a laboratory extraction procedure (referred to in this report as EP-III) can be selected to simulate the leaching of an industrial waste co-disposed with municipal wastes in a landfill. The intended characteristics of EP-III include the following:

1. Capable of simulating leaching in a landfill containing municipal and industrial wastes in proportions of about 95 and 5% by weight, respectively.
2. Compatible with biological toxicity tests (e.g., mutagenic, aquatic, and phytotoxic).
3. Relatively inexpensive to conduct in terms of time, equipment, and personnel.

STRATEGY

The strategy used for the development of EP-III was as follows:

1. Large-scale field lysimeters filled with municipal waste were used to generate a MWL.
2. This MWL was then used to leach selected industrial wastes under simulated landfill conditions.

3. The concentrations of inorganic and organic constituents observed in the industrial waste leachates (in excess of the control MWL) were plotted relative to their liquid/solid ratio (i.e., the volume of leachate divided by the weight of the waste).
4. A variety of laboratory methods (combinations of extraction procedures and media) were used to produce extracts.
5. The preferred laboratory extraction method was determined by comparing the concentrations of the inorganic and organic constituents in the laboratory extracts to five sets of target concentrations established to simulate various leaching scenarios.

METHODS AND MATERIALS

FIELD LYSIMETERS

About 1.5 Mg of municipal waste obtained from the city of Oak Ridge, Tennessee, were packed in each of the two large-scale "lysimeters" (lined concrete cylinders 1.8 m in diameter and 3.6 m in height; Fig. 1). The municipal waste consisted of a residential waste (collected from households) and a commercial waste (collected from the downtown shopping area and fast-food restaurants). The lysimeters were filled February 26, 1982, in the manner illustrated in Fig. 2. A clay loam soil was added to the top of the waste to prevent moisture and vapor loss. About 750 L of water was added to each lysimeter within 5 d of filling the lysimeter, to provide moisture for microbial degradation of the waste. During the filling of the lysimeters, no attempt was made to simulate the packing density of a commercial landfill. By August, the waste had settled to about 1.5 m below the top of the lysimeter, a density of 280 kg/m^3. This figure is still significantly below the design density of a commercial landfill (about 500 kg/m^3; Hagerty et al. 1973). However, the density compares closely with the municipal waste test cells 300 kg/m^3 constructed by Walsh et al. (1981).

On June 3, 1982, the pumping of distilled water to the lysimeters began (see Fig. 3 for a flow diagram). Ten pumps and flow meters (five for each lysimeter, Fig. 4) were used to ensure that the desired flow rate of 26.5 L/d in each lysimeter) was attained. Leachate from the lysimeters was diverted to the overflow collector until August 6, 1982 (i.e., 64 d elapsed time). On this date the leaching of the selected industrial wastes began. Operation of the lysimeters from June to August 6, 1982, was intended to allow sufficient time for a steady-state to be obtained between the leachate flow and decomposition rate of the municipal waste. The objective was to generate a MWL that did not vary significantly in its physicochemical characteristics over the duration in which it was to be used as a

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leaching medium. Major changes in MWL characteristics would likely result in significant changes in leaching of the various constituents from the industrial wastes. Hydraulic residence time for each of the lysimeters was an estimated 30 to 40 d.

Leachate from each of the lysimeters was collected in 30-L glass carboys that served as sumps for two recycling pumps (10 to 15 mL/min) to the glass manifolds (Fig. 5). Excess leachate flowed through a check-valve (which kept the leachate anoxic) to an overflow collecter (Fig. 3). The glass manifolds (Fig. 6) were used to divert MWL to columns containing the industrial wastes and to support the electrodes necessary to monitor pH and Eh (a combination glass pH and Ag/AgCl reference electrode and a Pt electrode). Flow at 0.8 mL/min of MWL to each of the industrial waste columns was accomplished using two 15-channel peristaltic pumps (Fig. 6). All tubing used to transport MWL was made of polytetrafluoroethylene (PTFE) except the short length of peristaltic pump tubing, which was a silicone composition. The 40 borosilicate glass columns (38.7 cm i.d. by 30.5 cm in height) used to contain the industrial wastes were supported on a wooden frame (20 columns each on the top and bottom racks) in front of the lysimeters (Fig. 7). The columns were covered with a 2-cm thick plexiglass plate which had the underside covered with an overlay made of PTFE to protect the plexiglass plate against (1) decomposition by organic solvents and (2) contamination of the resulting leachate with organic compounds solubilized from the plexiglass plate. Wastes were layered between 5 to 7 cm of sand (Fig. 8). Sand was used to prevent entrainment of the waste by the leachate and to distribute the MWL uniformly across the surface of the waste. Between the top layer of sand and the waste, a thin layer of glass wool was used to further facilitate lateral dispersion of the MWL. Two quantities of waste were used, 3.6 kg in the top 20 columns and 0.8 kg in the bottom 20 columns. The different quantities of waste represented two separate experiments. Leachate from the top 20 columns was sampled twice weekly the first 30 d and once weekly for the remainder of the experiment (79 d of leaching). The intent was to leach the wastes until a liquid/solid ratio of 20:1 was obtained (similar to the ratio now being used in the EP). Columns on the bottom rack were sampled once after 28 d of leaching (also equivalent to a 20:1 liquid/solid ratio). Thus, analyses from the top columns represented leachate concentrations over time, while analyses from the columns on the bottom rack represented an integrated leachate concentration over the duration of the experiment (in this case 28 d).

Leachate was collected from the industrial waste columns in 15- and 20-L Tedlar bags. Tedlar bags, made from polyvinylfluoride, are known to preserve the integrity of air samples collected for volatile organic compounds (Schuetzle et al. 1975). Because diffusion in a liquid is much slower than in a gas, they were expected to be very good sample containers for all analytes. The larger bags were used for the 28-d experiment in which a single sample was taken, and the smaller bags (15 L) were used for the 79-d experiment in which samples were taken weekly and biweekly. In the latter experiment, any excess leachate after sampling was discarded and the same Tedlar bag was used for collection of the next sample. Use of these Tedlar bags allowed collection of volatile organic compounds and maintenance of an anoxic environment to minimize oxidation of reduced iron and formation of secondary precipitation products. Previous experiments had confirmed that the Tedlar bags would prevent losses from vaporization, minimize adsorptive losses of nonpolar organic compounds, and reduce precipitation of inorganic constituents by maintaining an anoxic condition. Depending on the wastes, aliquots were taken for analyses for volatile- and nonvolatile-organic compounds and inorganic constituents.

LABORATORY EXTRACTIONS

Laboratory extractions were conducted using rotary-batch and upflow-column methods. Rotary-batch extractions were made at liquid/solid ratios of 2.5, 5, 10, and 20:1 using a rotary extractor (equipped with six 2-L borosilicate glass vessels and rotating at 30 rpm) over an 18-h period (overnight). The 18-h extraction interval was selected for operational reasons (previous research had indicated most extracts are in chemical equilibrium by 18 h [Brown et al. 1983]). This would allow sufficient time to filter the extracts, clean the extractor vessels, and start a new set of samples in the same day. One-hundred grams of waste was used for all batch extractions except the 2.5:1 liquid/solid ratio in which 200 g of waste was used with 500 mL of extracting medium to obtain sufficient quantity of leachate for inorganic and organic analyses.

Upflow-column extractions were conducted using glass chromatography columns (2.5 cm i.d. and 45 cm in length) equipped with PTFE-tipped adjustable plungers to accommodate wastes of varying densities; see Fig. 9. A flow rate of about 1.4 mL/min was used to reach a liquid/solid ratio of 20:1 in 24 h for a 100-g sample of waste. An 18-h extraction would have been preferable (to allow comparison to the extraction time used with the rotary extractor), but such a flow rate (about 2 mL/min) was not permissible for many of the wastes in the 2.5-cm diameter column. For the electroplating waste, even when the waste was mixed with 100 g of sand (50:50 mixture), considerable pressure was encountered, resulting in longer extraction times. A relief valve, set at 4.1×10^5 Pa (60 psi), was used to avoid unsafe stress on the glass column. Mixing with sand was not required for the other wastes. Aliquots were taken at specific intervals of time to make liquid/solid ratios of 2.5, 5, 10, and 20:1. Thus, unlike the batch extractions, a single-column extraction was conducted with aliquots taken to correspond to the appropriate liquid/solid ratio (e.g., aliquots were taken at 250, 250, 500, and 1000 mL). Concentrations of the inorganic and organic constituents at the various liquid/solid ratios were calculated using the concentrations determined in the first 250-mL aliquot (2.5:1 liquid/solid ratio), the first and second 250-mL aliquots (5:1 liquid/solid ratio), etc.

In retrospect, because of the difficulties in maintaining adequate flow rates through the 2.5-cm-diameter columns for wastes of low hydraulic conductivity, a preferred column design would be a column of larger diameter. Recent experimental work with a 10-cm-diameter column loaded with 200 g of waste has indicated that such a design would eliminate this problem for most wastes. For example, the limiting hydraulic conductivity of such a column would be reduced by a factor of 8 (5.9×10^{-4} cm/s as compared to 4.7 to 10^{-3} cm/s). Purchase of such a column before starting the laboratory experiments was attempted; however, a vender could not be found that carried a 10-cm-diameter glass column with PTFE-tipped adjustable plungers. Use of the larger diameter column would significantly enhance the operational aspects; however, it is not anticipated that the characteristics of the extracts would differ measurably from those of the 2.5-cm-diameter column.

All extracts were filtered through a 0.4-μm polycarbonate filter (Nuclepore). Preliminary experiments that were conducted to assess the relative effectiveness of Nuclepore, Millipore, and Ag filters to retain organic compounds extracted from the wastes indicated no significant ($P < 0.05$) differences among filter type (Regan 1983). The column extracts were filtered via an in-line filter (47 mm i.d.) fitted with special PTFE gaskets. The rotary-batch extracts were filtered using a Millipore pressure filtration apparatus and 142-mm diameter filter disks at $\leq 5.1 \times 10^5$ Pa (75 psi). For waste samples that contained volatile organic compounds, the column effluents were collected in 5-L Tedlar bags directly connected to the in-line filter.

The extracting media were: (1) pH 5 sodium acetate (0.1 $\underline{M}$ with respect to acetate; this is equivalent to the maximum acetate added in the EP), (2) a weak carbonic acid solution (carbon-dioxide-saturated de-ionized distilled water), (3) de-ionized distilled water, and (4) fresh ORNL/MWL from the lysimeters. The ORNL/MWL was collected in a Tedlar bag immediately before use as an extractant to maintain its anoxic status. For the upflow-column extractions, the MWL was pumped directly from the Tedlar bag. For the rotary-batch extractions, fresh anoxic MWL was added directly to the preweighed waste and then immediately extracted. No attempt was made to maintain an anoxic environment in the batch extractions other than adding the fresh MWL. The carbonic acid solution was produced by bubbling carbon dioxide gas (99.8% purity) through a fritted glass dispersion tube in de-ionized distilled water for 18 h before using it as an extracting medium. The solution was added directly to the preweighed wastes in the extraction flasks; the extraction flasks were immediately closed and extraction was initiated. As with the rotary-batch extractions with MWL, no attempt was made to maintain an anoxic environment during extraction. For example, there was no attempt to continue the bubbling of carbon dioxide gas into the waste suspension while the rotary-batch extraction was in process. In the upflow-column extractions, however, the supply of influent solution was continually purged with carbon dioxide gas throughout the extraction period. Thus, upflow-column extractions by the MWL and carbonic acid extracting media were maintained in an anoxic status, while the rotary-batch extractions were anoxic only to the degree of the poising capacity of the MWL and carbonic acid.

Rationale for Selection of Extraction Procedures and Media

Laboratory extraction tests for solid wastes have been studied extensively over the past few years. Lowenbach (1978) provided a detailed summary of test procedures for over 30 proposed leaching tests. A comprehensive evaluation of the effects produced by systematically varying leaching test conditions has been conducted by Ham et al. (1978, 1979a, and 1979b). The intent of this research was to compare a variety of extraction media, extraction procedures, and varying liquid/solid ratios to determine the preferred method to simulate concentrations of inorganic and organic constituents in lysimeter leachates.

Earlier research (Brown et al. 1983) showed that the upflow-column extraction procedure was superior to rotary-batch extraction for removal of trace levels of nonpolar organics from a variety of solid wastes. Column extractions also provide dynamic data that batch extractions do not. From a physical sense (liquid/solid ratio), column extractions imitate leaching in a landfill better than batch-type extractions that have to be carried out at liquid/solid ratios of 5:1 and greater. Batch extractions, on the other hand, tend to be much easier to conduct from an operational sense than column extractions. Because no pumps are needed for batch extractions, they are less costly and extraction time is not dependent on hydraulic conductivity of the waste, as it is with column extractions. Batch extractions tend to be less complicated to perform; thus, less training of laboratory personnel is required, and variability between operators is less likely to occur than with column extractions.

The 0.1 M acetate pH 5 buffer solution was selected because (1) it contains the most dominant carboxylic acid in MWL (it is also similar to the EP in respect to maximum acetate concentrations), (2) the low pH simulates an acidic MWL environment, and (3) operationally the medium is easier to use than the EP, which requires initial titration and periodic adjustments to pH 5. The carbonic acid medium was selected because it provides an acidic medium and does not add any inorganic or organic constituents that confound the character of, or interfere in, the biotesting of the final extract. Water was selected as a medium because (1) it is the universal medium, (2) it does not add any inorganic or organic constituents that would interfere with biotesting of the final extract, and (3) water has been used as a leaching medium for many leaching procedures (Lowenbach 1978). Municipal waste leachate (ORNL/MWL) was used as a laboratory extractant because it most closely resembles the extraction medium at the lysimeters. The original intent was to reconstitute a synthetic MWL from the various carboxylic acids and the dominant inorganic constituents such as Ca, Mg, Na, and K as measured in the ORNL/MWL.

Because laboratory extraction was being conducted concurrently with the lysimeter leaching, the opportunity was taken to test whether the same extracting medium (ORNL/MWL) would extract waste constituents in a similar manner under laboratory conditions as under field conditions. The major difference was that the laboratory extraction was an 18-h test of 79 d of leaching.

WASTES SELECTED

Four wastes were used in the field and laboratory studies. One waste, the heavy ends and column bottoms from the production of tri- and perchloroethylene (henceforth called dichloroethylene still bottoms), was predominantly organic. Two wastes, one a paint production sludge and the other a mixture of petroleum refining incinerator ash and an American Petroleum Institute (API) separator sludge, contained both inorganic and organic hazardous constituents. The fourth waste, an electroplating wastewater treatment waste, was primarily inorganic in character. These wastes were selected in collaboration with U.S. Environmental Protection Agency, Office of Solid Waste.

Sawdust was added to the dichloroethylene still bottoms and the paint sludge because these wastes contained considerable free liquid. This was the disposal practice used by the manufacturer of the paint sludge. Sawdust, clays, and other sorbing-type materials are often added to liquid wastes for solidification, stabilization, and fixation purposes. Thus, admixtures of sawdust made to adsorb the free liquid of the dichloroethylene still bottoms and paint sludge were used in the field and laboratory studies. Air-dried sawdust was added to the wastes in amounts large enough to effectively sorb the liquid component of the waste. The proportions of sawdust used are presented in the section on experimental design of the field lysimeters.

The physicochemical characteristics of the wastes (and the admixtures made from the wastes and sawdust) and inorganic elemental concentrations (except dichloroethylene still bottoms) are presented in Table 1. The methods used in the dissolution of the wastes and the analyses of inorganic and organic constituents are presented in the Chemical Analyses section. The solids content was determined after drying for 24 h at 90°C. The porosity represents the quantity of interstitial water held by the waste under simulated field conditions. This was determined from the weight loss at 90°C for 24 h (expressed as percent of total wet weight) on a sample of waste taken from a downward-flow column operated at a flow rate equivalent to that used in the field lysimeter experiment (7 mL/min/m^2).

Analyses for organic compounds in the API/incinerator ash, dichloroethylene still bottoms, and paint sludge revealed a variety of organic compounds (Table 2). Again, the methodology used to isolate and measure these organic compounds is presented in the Chemical Analyses section. The elemental concentrations measured in EP extracts are presented in Table 3. Note that only one of these wastes, the API/incinerator ash, would fail the EP toxicity test criterion (because of Cr concentrations; see Table 3). Chromium concentrations reported in Table 3 are total Cr concentrations; however, other extractions of the API/incinerator ash revealed that well over 50% of the extractable Cr is hexavalent. Extraction of the organic constituents from the wastes using the EP protocol are presented in Table 4.

EXPERIMENTAL DESIGN

Field Lysimeters

Two separate experiments involving the same wastes were conducted at the field lysimeter site. The major differences in the experiments were the quantity of waste used (3.6 and 0.8 kg), the duration of leaching (79 and 28 d), and the method of sampling (periodic and single-sample dates). The objective of the longer experiment (the one in which 3.6 kg of waste was used) was to determine the concentrations, in terms of liquid/solid ratio, of the organic and inorganic constituents observed over the 79-d leaching period. The quantity of waste (3.6 kg) also roughly approximates the desired 95 to 5% (municipal/industrial) co-disposal scenario mentioned earlier. The objective of the 28-d experiment was to evaluate the effectiveness of MWL to leach organic and inorganic constituents from the industrial wastes, relative to those extracted by the EP toxicity test. For the sake of simplicity, the 79-d experiment will be called "Target" and the 28-d leaching experiment, "Aggressor." Wastes used in the lysimeter experiment included: (1) an admixture of API separator sludge and refinery incinerator ash (respectively 28 and 72% by weight), (2) dichloroethylene still bottoms mixed with sawdust (respectively 28 and 72% by weight), (3) liquid and bottom fractions of a paint production sludge mixed with sawdust (respectively 33, 33, and 33% by weight), and (4) an electroplating wastewater treatment sludge (used as received).

Laboratory experiments were conducted to ascertain the desired proportions of each admixture component. The criterion was addition of the liquid component until free liquid emerged from the bottom of the laboratory test column. All wastes were rolled on a drum-roller prior to mixing. Mixing of the admixtures (for each column) was conducted in 19 L PTFE lined buckets.

MWL from each of the two lysimeters was pumped to two columns containing each of the industrial wastes. Thus, each experiment included four industrial waste columns for each waste, making a total of 16 test columns (2 lysimeters x 4 wastes x 2 replicates). Four additional columns, two containing only sand and two others containing sand and sawdust, were used as controls. The columns containing these treatments were randomly assigned positions on the support rack in front of the lysimeters (Fig. 7). Leachates from the columns containing the four industrial wastes were collected as described earlier. To ensure that the total quantity of inorganic and organic constituents leached from the wastes was detected, analyses were made on unfiltered leachate samples. Inorganic elemental analyses were also conducted on filtered leachates. Filtration was carried out using 0.4- m (nominal pore size) polycarbonate filters (Nuclepore) and vacuum filtration. This work was undertaken to evaluate the influence of the secondary precipitation that took place between sampling dates (even in the relatively anoxic environment of the Tedlar bags). Samples of filtered and unfiltered leachate were preserved by acidifying to pH 2 (USEPA 1979).

Organic analyses of leachates from the field lysimeter facility reported herein are those determined on unfiltered leachates. All ICP inorganic analyses of lysimeter leachates, unless otherwise specified, are average concentrations determined on filtered and unfiltered leachates. On the other hand, all AAS inorganic analyses, to determine elemental NIFDWS concentrations that were below ICP detection limits, were determined on filtered leachates (0.4-μm polycarbonate Nuclepore filters).

Laboratory Extractions

Laboratory extraction studies consisted of two procedures, upflow-column and rotary-batch, and four extraction media: (1) sodium acetate pH 5 buffer (0.1 M with respect to acetate; this is equivalent to the maximum acetate added in the EP), (2) carbon-dioxide-saturated de-ionized distilled water (weak carbonic acid solution; pH about 3.5), (3) de-ionized distilled water, and (4) fresh MWL (ORNL/MWL) from the field lysimeter, collected in Tedlar bags to maintain the leachate in an anoxic condition. These two procedures with each of the four media were conducted at four liquid/solid ratios (2.5, 5, 10, and 20:1) with two replications for a total of 256 aliquots (4 wastes × 2 procedures × 4 media × 4 liquid/solid ratios × 2 replicates). Two ancillary extraction methods were also included: (1) the RCRA-approved EP and (2) a bi-sequential extraction method. The bi-sequential method was developed to extract high concentrations of readily soluble constituents and acid-soluble metals from predominantly alkaline wastes. The method consisted of a rotary-batch procedure at an initial liquid/solid ratio of 5:1, followed by adding a fresh 100 g of waste to the filtered extract and extracting for another 18 h to make a final liquid/solid ratio 2.5:1. The extraction medium chosen was sodium acetate pH 5 buffer. For the electroplating waste, a preliminary experiment revealed that it was necessary to use a concentration of 0.25 M acetate to bring the pH of the final extract to values less than 7.5. For the other three wastes, 0.1 M sodium acetate pH 5 buffer was used.

Statistical Procedures

Statistical analyses were performed using the Statistical Analysis System (SAS) software package provided by SAS Institute (SAS 1982). An analysis of variance test was conducted on the data using the General Linear Models (GLM) procedure as outlined in Chapter 9 of SAS (1982). A Duncan's multiple-range test was then used to determine whether these were significant differences among treatment means.

CHEMICAL ANALYSES

Chemical analyses of the four wastes indicated potential contaminants in their solid waste leachates. Before any field or laboratory work was started, tentative "target" constituents for each waste were identified. Leachates and extracts from these wastes were analyzed for these target constituents over the duration of the experimental work. The target constituents for the four wastes were as follows:

API/Incinerator Ash
- Inorganic elements - Cr, As, Cd, and Pb
- Organic compounds - Naphthalene

Dichloroethylene Still Bottoms
- Inorganic elements - None
- Organic compounds - Dichloroethylene, Trichloroethylene, Dichloroethane, Trichloroethane, Toluene, Xylenes, and Hexachlorobutadiene

Paint Sludge
    Inorganic elements - Ba, Cr, and Pb
    Organic compounds - Ethoxyethanol, Ethoxyethyl Acetate, Toluene, and Xylenes

Electroplating Waste
    Inorganic elements - Pb and Cr
    Organic compounds - None

The leachates were analyzed for target inorganic elements using atomic absorption spectroscopy (AAS). The leachates were also analyzed on a routine basis for additional inorganic elements using inductively coupled plasma (ICP) spectroscopy to measure the leaching of the waste's matrix elements (e.g., Ca, Na, K, Al, Fe) and to identify other toxic elements that might be useful as targets. The procedures used to analyze the total content of inorganic and organic constituents in the waste, as well as the procedures to measure those constituents in the leachates and extracts of those wastes, are presented below. For constituents below the detection limits, estimates of the concentration were reported as one half the detection limit rather than as the detection limit itself or as zero.

Organic Chemical Analysis of Wastes

The analysis of the wastes containing organic compounds was conducted in two phases. In phase one, the wastes were extracted and analyzed by gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS) to qualitatively identify potential target organic compounds. The criteria used for selection of target compounds were:

1. Presence in the waste at sufficient levels for detection in leachate.
2. Presence in ORNL/MWL at levels less than 5% of those in industrial waste leachates.
3. Availability of rapid, automated analytical methods.

In phase two, the waste extracts were quantitatively analyzed in triplicate using the methods described in the following sections and also using an independent method wherever possible.

Each waste was extracted using a Branson Model 185 Sonifier. A 1-g aliquot was added to a 25-mL centrifuge tube and diluted with 10 mL solvent. Sonication was performed for 15 min. The mixture was then centrifuged at 2000 rpm for 10 min, and 5 mL of supernatant was withdrawn. The extraction solvent was chosen to provide maximum dissolution of the sample while not interfering with the subsequent analyses. Methylene chloride was used for API/incinerator ash, methanol for volatile components of dichloroethylene still bottoms, methylene chloride for nonvolatile components of dichloroethylene still bottoms, and acetone for paint waste.

Semivolatile components were analyzed using a Hewlett-Packard (H-P) Model 5736A gas chromatograph equipped with flame ionization detection, a 30 m x 0.25 mm i.d. DB-5 fused silica open tubular column, and a Model 3390A integrator. GC/MS was performed using a H-P 5985A gas chromatograph/mass spectrometer detection system in the electron impact mode, with a 30 m x 0.25 mm i.d. DB-5 column. The injector temperature was 275°C, detector temperature was 300°C, and the oven was programmed from 40 to 250°C at 4°C/min. Compounds were identified by comparison with library spectra.

Volatile organic compounds were analyzed using a Perkin-Elmer 3920 gas chromatograph equipped with flame ionization detection and a 2 m x 3 mm o.d. glass column (1% SP-1000 on Carbopak B). GC/MS was performed using an H-P 5992 gas chromatograph/mass spectrometer equipped with an identical column.

API/Incinerator Ash: Open tubular GC analysis of API/incinerator ash extract (Fig. 10) revealed a complex mixture typical of oily sludges. The major compounds identified in API/incinerator ash by open tubular GC/MS included: toluene, xylene, ethylbenzene, nonane, nonene, decene, C_3-benzenes (3 isomers), decane, C_4-benzenes (12 isomers), undecane, C_5-benzenes (4 isomers), naphthalene, dodecane, tetradecane, methylnaphthalene, biphenyl, C_2-naphthalenes (4 isomers), and C_1-biphenyl.

Many other constituents could be generically identified as higher alkyl benzenes, alkyl phenols, and branched aliphatic hydrocarbons. Interestingly, no higher molecular weight polynuclear aromatic hydrocarbons (PAH) were found, even though the presence of these compounds might have been anticipated. Phenol was detected, but at a very low level (0.83 mg/kg). None of the components was present at greater than 100 mg/kg (tetradecane was found at 95 mg/kg). Most of the constituents could not be detected above the background of MWL and were, therefore, inappropriate as target organic compounds.

Compounds selected for tentative target organic analysis included toluene, xylene, and naphthalene (Table 2). Quantitative analysis was then performed in triplicate. The results were as follows: the average value of naphthalene (20.5 mg/kg ± 1.2) by high-pressure liquid chromatography (HPLC) compared well with the level found by GC (23.4 mg/kg) and that found by GC/MS (19.2 mg/kg). The higher GC value indicates the presence of interferences coeluting with naphthalene.

The levels of toluene and xylene found in industrial waste/MWL leachates were considered too low to be useful as target compounds, as 1 to 2 mg/L of these compounds was also present in MWL. The naphthalene level was considered useful, because naphthalene was not found in MWL and a sensitive, selective method was available (HPLC-fluorescence).

Dichloroethylene Still Bottoms: Qualitative analysis of the volatile organic compounds present in dichloroethylene still bottoms revealed the presence of a range of halocarbons, including methylene chloride, cis and trans 1,2-dichloroethylene, 1,1,1-trichloroethane, chloroform, trichloroethylene, tetrachloroethane, tetrachloroethylene (unresolved pair), 1,1-dichloropropane, and 1,2-dichloropropane. Because all these halocarbons were expected to behave similarly in extraction experiments, only the major constituents were quantitated for use as target organic compounds (Table 2). All compounds were considered candidates as target organics, although the precision of the determination was not particularly good. The lack of precision occurs primarily because of the high vapor pressure of these compounds and the potential for evaporative losses during extraction and analysis. For example, recovery of these compounds added to the sawdust was approximately 60%. Thus, the quantity of compounds classified as "volatile" must be considered an estimate, and should not be used as an absolute concentration for mass balance calculations.

The semivolatile constituents of dichloroethylene still bottoms were analyzed as well. Qualitative analysis of major constituents identified included tetrachlorobutadiene isomers, pentachlorobutadiene, and hexachlorobutadiene (HCB) (Fig. 11). Because about 85% of the mass of the extract (peak area measurement using electronic integration) was accounted for by HCB, this compound was selected as a target organic compound. The quantity present in the sawdust admixture of dichloroethylene still bottoms was 224 ± 26 mg/g (n = 3).

Paint Waste: Qualitative analysis of the paint waste revealed that the organic components present were all relatively volatile solvents, including 2-ethoxyethanol, benzene, toluene, 2-ethoxyethylacetate, xylenes, and C_3-benzenes. An additional component was identified (tentatively) as a polymer of molecular weight 500. This component was not considered useful for target analysis because of the vagaries in its composition. However, early extracts from the lysimeter experiment were analyzed. No evidence was found for the migration of this component from the waste; therefore, no further work on characterization or determination was carried out. Quantitative analysis of the solvents mentioned earlier yielded the results in

Table 2. However, recovery of labelled toluene was only 46% from the sawdust admixture.

Benzene was not considered useful as a target organic compound because of its moderately low level (50 mg/kg) and the presence of analytical interference in MWL. C_3-benzenes were not useful because several unresolved isomers were present, causing problems in quantification. Furthermore, the behavior of these compounds was anticipated to be substantially similar to that of the xylenes.

Electroplating Waste: Organic analysis of electroplating waste did not reveal the presence of any potential target organic compounds.

Inorganic Chemical Analysis of Wastes

Each waste was analyzed directly by neutron activation analysis (NAA) and, after digestion, by ICP spectrometry. The two independent measurements were used to obtain accurate estimates of the total mass of inorganic materials in each waste. These two methods have been used previously to characterize the inorganic elemental content of other wastes (Griest et al. 1983).

The ICP spectrometer simultaneously measures the concentration of as many as 34 elements in solutions. The high temperature argon plasma source provides energy to cause characteristic optical emission from elements present. The intensity and pattern of these emission energies are related to concentrations of each element that are resolved by the optics in the spectrometer system to generate output in concentration of each element. The total measurement time is less than 5 min/specimen. The precision and accuracy obtained at the 1 mg/L concentration level, shown in Table 5, is about ± 10% at the 95% confidence level.

For NAA, the samples are irradiated twice. The first time they are irradiated for 20 s to determine uranium by the "Delayed Neutron Counting Method" (Dyer et al. 1962) and to determine, by gamma-ray spectrometry, those elements that have isotopes with half-lives ranging from 1 min to 3 h. The sample is then re-irradiated for 30 min and allowed to decay for 2 to 3 d. A gamma-ray spectrum is obtained to determine elements with half-lives in the range of 12 h to 3 to 4 d. After 2 to 3 weeks decay, elements with half-lives from 5 to 6 d to years are determined.

Absolute NAA uses isotopic abundances, neutron cross-sections, half-lives, and decay schemes to calculate the concentration of the elements. These physical constants are known, with uncertainties ranging from 0.001 to 10 and 15%. An overall uncertainty of ± 6% is used. Counting statistics ranged from 0.1 to 30%, depending on the concentration of the element and the sample matrix.

For ICP analysis, the wastes were completely dissolved using a lithium borate fusion technique. The lithium borate fusion technique retains virtually all elements except mercury. Partial loss of some volatile nonmetals (As, Se) may occur, and further study is under way to determine the extent of such loss.

The specimen to be fused should have a particle size of less than 150 mesh. In this work, specimen weights of 100 mg and a 500-mg portion of lithium borate were used. Smaller or larger specimen weights may be used, but the 5:1 ratio of lithium borate to specimen must be used. The lithium borate was placed in a pure graphite crucible, the waste was added, and the two powders were mixed using a small platinum wire or spatula. The covered crucible was heated in a muffle furnace at 950°C for 10 min and cooled. The crucible lid was removed and the crucible was reheated with occasional swirling for 10 min. The hot crucible was removed, and the melt was poured rapidly into a 50-mL beaker containing 40 mL of 3% nitric acid. The beaker was agitated until solution of the melt was complete. The solution was diluted to appropriate volume, and the ICP spectroscopic analysis was performed. The procedures are quality assured using National Bureau of Standards (NBS) standard reference materials and

United States Geological Survey (USGS) standard rocks. The results obtained by fusing the NBS standard reference materials 1633A fly ash mineral with lithium borate and then analyzing the melt solution in 3% nitric acid are shown in Table 6. The instruments used were:

NAA: Oak Ridge Research Reactor, coupled ND-6620 data acquisition system using lithium germanium detectors.

ICP: Instruments SA model JY-48 with 34 element channels. The power source is a Plasmatherm 2.5 kW (max) generator.

The results of the inorganic analyses of the wastes and admixtures of the wastes used in this study are shown in Table 1. Both ICP and NAA analyses were performed on five elements. The comparative data are shown only for Na, Mg, Ca, Cr, and Mn. The agreement is quite good both at high concentrations (Ca in API/incinerator ash) and low concentrations (Mn in paint waste). Because the object of the comparison was to ensure the reliability of the digestion technique and the ICP measurements, the level of comparability was considered adequate. Mass balance data for the lysimeter were generated using ICP data.

Methods for Organic Analysis of Leachates

Carboxylic Acids in Municipal Waste Leachate: Several methods exist for determining low molecular weight carboxylic acids in water, but each method has limitations. Direct aqueous injection GC (Ho et al. 1976) is perhaps the most commonly used technique, but it is limited in terms of selectivity and sensitivity. Isolation of the acids on diethylaminoethyl-Sephadex (Sheffer 1965), followed by derivation and GC analysis (USEPA 1980), provides increased sensitivity and selectivity, but is tedious and requires a prior estimate of the level of acids present. Solvent partition followed by GC analysis provides only limited improvement in terms of selectivity and sensitivity, and the analytical columns normally used are of limited durability. The method developed for use in this work involves reverse-phase HPLC coupled with conductivity detection. This method was shown to have significant advantages over the methods mentioned earlier, particularly in terms of selectivity (Manning and Maskarinec 1983).

Formic, acetic, propionic, butyric, valeric, hexanoic, heptanoic, and octanoic acids (C_1 through C_8) were obtained from the Aldrich Chemical Company, and aqueous standards were prepared without further purification. Reagent grade diethylether (J.T. Baker Chemical Co.) was used for the extraction procedure. The conditions used for the analysis were:

System: Two Waters Associates (Milford, Massachusetts) Model 6000A chromatography pumps, a Waters Associates WISP 710A sample processor, and a Waters Model 720 system controller.

Column: Zorbax-ODS, 7 μm particles, 25-cm x 15-mm o.d. Flow rate 1 mL/min.

Mobile Phase: Batch gradient:water; 50/50 water/methanol.

Detection: A Wescan Model 212 Conductivity Detector.

Peak ID and Quantitation: Manual measurement, using a H-P Model 7045A X-Y recorder.

The diethylether extraction/concentration was carried out by transferring an aliquot of the aqueous sample, usually 2 to 10 mL, to a 50-mL beaker and adjusting the pH to 2 ± 0.2 by the dropwise addition of 1 N sulfuric acid. The solution was transferred to a separatory funnel and extracted twice with 10- to 15-mL portions of ether. The mixture was shaken for about 4 min for each extraction.

A 4-mL aliquot of doubly distilled water was added to the combined ether extracts contained in a 50-mL beaker, and the beaker was placed in a ventilated hood to allow the ether to evaporate (25 min) over the distilled water. The acid isolate was then adjusted to a known volume (usually 5 mL), and an aliquot was injected into the column. The recovery was measured by spiking authentic standards into distilled water and carrying the water through the extraction/concentration step.

The method used in this work involves a solvent partition extraction concentration step followed by separation of the carboxylic acids by reverse-phase HPLC and conductivity detection. Initially, it was anticipated that MWL samples could be analyzed directly. However, MWL samples containing inorganic salts produced a "solvent peak" that completely obscured the formic and acetic acid peaks. Therefore, solvent partition was used as a means of excluding these interferences and also as a means of concentrating the MWL sample before analysis. Diethylether was chosen as the organic solvent because of its high volatility, good partition characteristics, and ability to exclude the inorganic interferences. Recovery of standard solutions of the various acids when carried through the extraction concentration procedure is shown in Table 7. The recovery was measured by comparing the areas of the peaks to those of the standard compounds. The sensitivity of the method is approximately 10 mg/L acetic acid and 100 mg/L octanoic acid in the final isolate. To improve the accuracy of the analytical procedure, authentic standards were prepared to match closely the MWL samples and were carried through the same extraction/concentration procedure as the samples. These extracted/concentrated standards were then used for calibration (external standardization).

The chromatograms obtained from authentic standards of the C_1 to C_8 carboxylic acids are shown in Fig. 12. The C_1 to C_4 acids are eluted using water as the mobile phase. Even this weak mobile phase does not provide for retention of the formic acid, although an estimate of formic acid concentration can still be made. The retention times of the heptanoic and octanoic acids are inordinately long using water, however, and it was more expedient to reinject the sample at a mobile phase strength of 50:50 water/methanol. Gradient elution was of limited usefulness because of the baseline drift of the conductivity detector as the mobile phase methanol concentration was varied. Acids of higher molecular weight than octanoic acid showed diminished response in conductivity detection. The asymmetric peaks result from the low capacity of the octadecylsilane (ODS) column for such polar compounds, although this does not affect the precision of the determination, which is ± 13%.

An example of the analysis of the MWL is shown in Fig. 13. Even though the MWL is an exceedingly complex mixture, the acids are detected with little interference. Special measures taken to ensure quality control were (1) analysis of a standard solution of the analytes with each set of five samples and (2) periodic analysis of a method blank.

Volatile Organic Compounds in Leachates: Methods for measuring volatile organic compounds in aqueous systems generally require some form of purge-and-trap GC (USEPA 1979). This involves purge of the aqueous sample with inert gas, collection of volatile organic compounds on a polymeric adsorbent (e.g., Tenax-GC), and subsequent thermal desorption followed by GC analysis. This method was not appropriate for the volatile organic compounds described in this work for several reasons. First, MWL foams excessively during purging, making the technique highly unreliable. Second, polar, water-soluble compounds such as ethoxyethanol do not purge efficiently at room temperature. Finally, the presence of nonvolatile components such as HCB contaminates the purge-and-trap system and requires long post-analysis bakeout times. Fortunately, the levels of volatile organic compounds in the leachates from the dichloroethylene still bottoms and paint sludge were high enough that direct aqueous injection of the samples was possible.

The volatile halocarbons (trans 1,2,-dichloroethylene, 1,2-dichloroethane, 1,1,1-trichloroethane, and trichloroethylene) were obtained in solution (0.12 mg/L) from Supelco, Inc., Bellefonte, Pennsylvania. Xylene was obtained from Mallinkrodt, St. Louis, Missouri; 2-ethoxyethanol from Aldrich, Milwaukee, Wisconsin; toluene from Burdick and Jackson, Muskegon, Michigan; and 2-ethoxyethyl acetate from MCB Regents, Cincinnati, Ohio. A stock solution of halocarbon mixture was prepared at 80 mg/L in methanol and used for calibration. A mixture of the paint solvent constituents was prepared at 100 mg/L. Both solutions were stored in the dark at freezer temperature. Samples were collected in septum-sealed vials with no headspace and were refrigerated until analysis. Separate instruments were used for the analysis of dichloroethylene still bottoms extracts and paint waste extracts. The operating conditions for analysis of volatile organic compounds in leachates from the dichloroethylene still bottoms and paint sludge are presented in Table 8.

Each calibration standard was analyzed at the beginning of the day. Therefore, no more than six samples were analyzed between calibrations. Table 9 lists the overall accuracy and precision data for each compound over the entire period of the study. Although the precision is not particularly high, the only consistent trend or bias noted was with 2-ethoxyethyl acetate. Daily recalibration ensured that the levels measured were more precise than the overall precision estimate suggests.

All media were analyzed with and without fortification of the target analytes. Dichloroethylene still bottoms constituents were fortified at 80 mg/L and paint sludge constituents at 100 mg/L. Toluene was detected in the MWL at approximately 1 mg/L. The MWL background was used as the detection limit for toluene. No other interferences were noted. Tables 10 and 11 include data on the recovery of each compound from each medium. Quality assurance measures taken were (1) daily calibration (recalibration, etc., once for each six samples), (2) regular analysis of blanks, and (3) analysis of blank media fortified with analytes.

Naphthalene in Leachates: Naphthalene is a nonpolar, aromatic organic compound readily extracted from water by methylene chloride. The method used in this work consisted of solvent partition with methylene chloride, concentration in a Kuderna-Danish evaporator, and analysis by reverse-phase HPLC using both ultraviolet (UV) (254 nm) and fluorescence detection and external calibration. Sensitivity of the method is 0.01 mg/L, reproducibility is ± 10%, and recovery is estimated to be 86% or greater over 0.01 to 1 mg/L MWL.

Naphthalene was obtained as part of a multicomponent PAH calibration mixture in methanol/methylene chloride (50:50) solution (Supelco, Inc., Bellefonte, Pennsylvania). Stock calibration solutions were prepared at levels of 40 and 4 mg/L. Aqueous extracts (500 mL or available volume) were adjusted to pH 12 with NaOH and extracted three times with 1/3-volume distilled-in-glass methylene chloride. The combined extracts were dried by pouring though a column of anhydrous sodium sulfate (50 mm x 30 mm i.d., previously cleaned with methylene chloride). The extracts were then concentrated to a volume of 1 mL using a Kuderna-Danish evaporative concentrator (Kontes, Vineland, New Jersey) equipped with a 5-mL receiving flask. The 1-mL concentrate was divided between two autosampler vials and analyzed twice by HPLC. Naphthalene in leachates was determined using the following conditions:

System: Beckman microprocessor-controlled Model 334 Gradient Liquid Chromatograph.

Column: Zorbax-ODS, 7-μm particles, 25-cm x 15-mm o.d. Protected by Brownlee precolumn cartridge. Flow rate 1 mL/min.

Gradient: 80% methanol/20% water for 10 min. Linear gradient, 90% methanol/10% water for 40 min.

Injection: 20-μL automatic injection with Altex valve, air-actuated, under controller command.

Peak ID and Integration: Manual ID of peak based on retention time of naphthalene standard injected closest in time to the sample. Peak height measured using H-P 3390A integrator.

Detection: Simultaneous UV (254-nm) and fluorescence (filter, 280-nm excitation, "blue from fluorescamine" emission). Integration performed using fluorescence detector.

To determine the validity of the analytical method with respect to MWL analysis, several experiments were carried out. A naphthalene spike was added to MWL at levels of 0.1, 0.2, and 1 mg/L. The MWL was analyzed, and recovery of naphthalene was determined to be 86% at the 1 mg/L level and 88% at the lower levels. This recovery was constant throughout the duration of the work (86.7% ± 3% at all levels, n = 15). There are two major causes of the lack of quantitative recovery. Naphthalene is soluble in water at the 30 μ g/L level; thus, complete partition does not occur. More important, naphthalene has appreciable vapor pressure and cannot be completely recovered during Kuderna-Danish evaporation. Under ideal conditions (methylene chloride spiked with naphthalene), 92% recovery was obtained.

Because the levels of naphthalene in MWL and laboratory extracts were considerably lower than those of other target organic compounds, MWL was considered to be a source of interference. Figure 14 shows a comparison of the naphthalene standard (0.08 mg/L equivalent MWL concentration), a MWL blank, and the MWL blank fortified with naphthalene (1 mg/L). Clearly, no interferences were seen in the MWL blank. Similar blanks were analyzed throughout the experiment, and none was found to contain detectable levels of naphthalene. In addition, each laboratory medium was analyzed and found to be free of contamination. Table 12 lists the recovery of naphthalene from each of the laboratory media after fortification.

Procedurally, each sample was injected twice, with a standard injected after each set of three samples had been analyzed. Coefficient of variation in the standard peak height from day-to-day was 15.7% (n = 14) at the 0.008 mg/L level and 5.5% (n = 11) at the 0.08-mg/L level. The readily apparent variations in the standard peak height are likely to have resulted from one or more of the following:

1. Presence of interfering compounds still adsorbed on the column.
2. Time since the column was completely cleaned.
3. Batch-to-batch variations of standard.
4. Minor variations in the baseline (integrator errors).
5. Drifting in the sensitivity of the detector.

Neither the condition of the standard nor that of the column is absolutely fixed, and the method of calculating naphthalene throughout this project reflects this. During the project, calibration standards were run 74 times (0.08 g/mL) and 17 times (0.008 g/mL). The values used for calibration were those obtained at the time of sample analysis. Variations over time resulting from the above factors were therefore minimized. Three quality assurance measures were taken:

1. Calibration standards were run once after each set of three samples.
2. A blank was analyzed for each set of 10 samples.
3. The calibration standard was periodically checked against an NBS-certified standard.

Hexachlorobutadiene in Leachates: HCB is a nonpolar organic compound readily soluble in cyclohexane. Because many polar organic constituents of MWL are not readily soluble in cyclohexane, this solvent was chosen for extraction of the HCB. Rinsing the cyclohexane extract with base, acid, and distilled water effectively excluded interferences while retaining 100% recovery. GC analysis was carried out using an automated gas chromatograph calibrated by external standardization.

The MWL or laboratory extract was analyzed as received. A measured volume (500 mL if available, less if not) was extracted three times with volumes of cyclohexane (distilled-in-glass grade). The cyclohexane extract was washed sequentially with pH 12 NaOH, pH 2 HCl, and distilled water. This was done to remove interferences resulting from the MWL itself. The cyclohexane was dried over anhydrous sodium sulfate that had previously been cleaned with methylene chloride. The extract was then diluted with cyclohexane to 40% of the volume of the original sample. Thus, a concentration factor of 2.5 was obtained. An aliquot of the cyclohexane extract was placed in an autosampler vial and injected into the gas chromatograph. The analysis conditions were as follows:

Calibration Standard: HCB (99%) obtained from Chem Service (West Chester, Pennsylvania).

Instrument: H-P Model 5840A equipped with H-P Model 7671 autosampler and flame ionization detector.

Column: 3.05-m × 0.32-cm o.d. glass, packed with 3% Dexsil-400 on Gas Chrom-Q.

Flow Rate: 50 mL/min He.

Temperature Program: 80 to 220°C, 8°/min.

Injection Temperature: 300°C

Detection Temperature: 320°C

Quantitation: Single-point external standardization at 40 mg/L using H-P 5840 data system.

With respect to the analysis of lysimeter leachates and laboratory extracts, MWL was considered to present the greatest challenge in terms of interferences and procedural difficulties. Therefore, a series of experiments was conducted to validate the method for MWL. Initially, a comparison was made of the extractability of HCB from MWL using both methylene chloride and cyclohexane. Samples fortified with 40 mg/L HCB were analyzed in duplicate using both solvents. The recoveries were not appreciably different, 98.9% for cyclohexane vs 98.6% for methylene chloride, but the cyclohexane extraction did not produce significant amounts of emulsion while methylene chloride did. Additionally, cyclohexane was much easier to dry than methylene chloride. For these reasons, cyclohexane was chosen as the extraction solvent.

Representative chromatograms of HCB, MWL blank, and MWL spiked with HCB (15 mg/L) are shown in Fig. 15. Clearly, no interference that could be attributed to MWL was seen. The linearity of the method was established over the range of 2.5 to 250 mg/L, and results are shown in Fig. 16. At all concentrations, recovery was essentially quantitative.

Each medium tested in the laboratory extractions was used to extract sawdust and then spiked with HCB to ensure that matrix effects would not occur. The results are shown in Table 13. Quality assurance measures taken were as follows:

1. Stock Solution: 100 mg/L in cyclohexane stored in the dark at freezer temperature and analyzed 4 times with each group of 8 to 12 samples.
2. Analytical Blanks: Distilled water carried through procedure and analyzed with each group of 8 to 12 samples.
3. Medium Blanks: MWL analyzed throughout lysimeter experiment. Each laboratory medium analyzed once.

Methods for Inorganic Analysis of Leachates

Inductively Coupled Plasma Spectroscopy: The ICP technique was used for multielement characterization of all extracts from both the lysimeter and the laboratory tests. MWL was used as the matrix for calibrating the instrument, using the standard addition method. The ORNL instrument is capable of simultaneous measurement of as many as 34 elements. The high temperature argon plasma source provides the excitation required to cause characteristic optical emission from the metals present. The optical system (lens, grating, photomultipliers) provides a voltage signal proportional to concentration for each element being measured. A PDP 11/23 computer converts the voltages to micrograms of element per milliliter of liquid specimen, corrected for interelement effects and spectral background interference.

The instrument is calibrated by measuring a series of carefully prepared and certified standard solutions under the same conditions used for analysis of routine specimens. The standards prepared to our specification (SPEX Industries, Metuchen, New Jersey) have been found generally satisfactory. Aqueous specimens in 3% nitric acid are normally required for analysis. Aqueous specimens are diluted to final volume with nitric acid. Interelement interference corrections are computed for each element present, using single-element standard solutions. The specific emission wavelengths measured were chosen to minimize both interelement interference and spectral background. A

Jobin Yvon (JY-48) ICP 34-channel polychromator, with a Plasmatherm 2.5-kW generator and PDP 11/23 controller-computer, was used as the analysis system. The initial calibration was as follows:

1. A two-point calibration curve for every element was determined by using 1-mg/L multielement SPEX standards, de-ionized water (zero), and a reagent blank (MWL).
2. After calibration, a representative sample was analyzed next with blank subtraction.
3. If all concentrations were less than 5 mg/L, interelement correction curves were determined.
4. If one or more elements were off-scale, the sample was diluted tenfold and reanalyzed. The dilution procedure was continued until a reasonable estimation of every concentration could be made.
5. Several samples from a group were analyzed in this way to obtain an idea of the maximum concentrations likely to be found in the group.
6. If it was not possible to keep all desired elements on scale with a single measurement, the photomultiplier tube voltages of the more concentrated elements were lowered.

Sample profiles were generated as follows:

1. Emission line profiles of a sample found to have the greatest concentrations and a blank were obtained with offset limits of ± 30 (entrance slit dial divisions) and an increment of three.
2. A printout of the profiles was used to determine background for off-peak corrections.
3. For those elements of interest, note that the background in the respective profile was in step. If it was flat or slightly sloped but quiet on one or both sides of the peak, a one- or two-point background correction was applied respectively. Background correction was used on noisy or steeply sloped backgrounds.
4. The best choice of background position is in the center of a flat region. Those positions that were common to most of the elements were used.
5. The background position for each desired element was entered in the table of the appropriate program.
6. If the background was the same in the blank and the sample, no correction was necessary.

A background-corrected calibration curve was determined using the 0- and 1-mg/L SPEX standards and background correction. One or more representative samples using these new curves were analyzed. All elements having concentrations approximately 1000 times or more than any element of interest were tested to determine whether they interfered with these elements. The interfering species were determined by beginning with the element that was most concentrated. High-purity single-element standards of this element, which bracket the expected concentrations in the samples, were used to make a new calibration curve for the element (using background correction if it was needed for other elements). The interelement correction curves were determined as follows:

1. The apparent concentration of all requested species of interest in each of the single-element standards (whether positive or negative) was noted.
2. Correction coefficients for all interfered elements were set up in the appropriate table based on the above information.
3. This procedure was repeated for all elements with high concentrations.
4. Interelement correction curves were not recalculated unless photomultiplier tube voltages or power was changed.

A standard spiked into MWL was analyzed (using background and/or interelement corrections if necessary) to determine the accuracy of the calibration curves. One or more samples were spiked with a multielement standard at the 1-mg/L level (more if the sample was high in all elements). If the average spike recovery was not 100 ± 10%, the dilution factor was used to correct the recovery. Deviations from a 100% recovery factor normally result from differences in acid concentration between the standard and samples. However, uniform acid concentration was obtained in this work.

Five to 10 mL of sample were placed in the autosampler test tubes. The tubes were arranged in the autosampler (from lowest to highest concentration, if possible) preceded by a blank. Samples were analyzed with the appropriate combination of blank subtract (B), dilution factor (D), and interelement corrections (K) in the concentration mode (P) with off-peak corrections, if necessary. Check standards, Environmental Protection Agency- (EPA-) or NBS-certified, were analyzed approximately every tenth sample and the ICP recalibrated if necessary. During the course of the study, 1065 samples were analyzed; thus, over 100 quality assurance controls were recorded.

Atomic Absorption Spectroscopy: All extracts analyzed for inorganic constituents were treated according to standard methods (USEPA 1979) and were directly analyzed for metals by flameless graphite furnace AAS, with the following exceptions:

1. Extracts for Hg determination were preserved by being added to a nitric acid/dichromate solution and worked up for cold vapor flameless AAS (Feldman 1974).
2. Se was chelated with 5-nitro-o-phenylene diamine and extracted with toluene before analysis (Talmi and Andren 1974).
3. As was determined by an arsine accumulation-helium glow detector procedure (Feldman 1979).

Samples were analyzed by the method of standard addition (USEPA 1979). Lead, Cd, and Cr were measured by a direct aspiration atomic absorption technique, and the furnace procedure was used if this technique did not provide adequate sensitivity.

Commercial single-element atomic absorption standards of 1 mg/L were obtained from Fisher Scientific Company for lead, cadmium, and chromium. Calibration solutions were prepared from these standards. Lead, cadmium, and chromium were analyzed by the direct aspiration or furnace technique described in USEPA (1979). The conditions used were as follows:

Furnace: Perkin-Elmer 603 Atomic Absorption Spectrophotometer 2200 Graphite Furnace, concentration calculated by standard addition. Optimum range: Pb and Cr, 5 to 100 µg/L; Cd, 0.5 to 10 µg/L.

Direct Aspiration: Perkin-Elmer 403 Atomic Absorption Spectrophotometer, concentration calculated from standard curve. Optimum range: Pb, 1 to 10 mg/L; Cd, 0.05 to 2 mg/L; and Cr, 0.5 to 10 mg/L.

For direct aspiration analysis, a standard curve was prepared in the optimum concentration range from the commercial standards. A curve was established at each analysis time using parameters described in the EPA methods. An EPA trace metal sample and an internal quality control sample were analyzed prior to sample analysis. During the analysis, the baseline was checked often, matrix interference was checked by spiking, calibration standards were checked frequently, and repetitive measurements were obtained.

Furnace measurements were made by standard addition. Standards, in the optimum concentration range, were prepared from the commercial standard. Instrument conditions described in USEPA (1979) were used. An EPA trace metal sample was analyzed prior to sample analysis. After an appropriate aliquot was determined, the sample was spiked twice. Appropriate background correction was made. Based on the analysis of internal controls and EPA samples, the accuracy of the measurements in the optimum concentration range by direct aspiration was within ± 5% of the true value. The accuracy of the furnace method using the above conditions was ± 10% of the true value. The relative standard deviation of repetitive measurements was estimated to be ± 5% for direct aspiration and ± 10% for furnace techniques at the above conditions. Near the detection limit, precision and accuracy decrease.

Ion Chromatography: A relatively new analytical technique, ion chromatography (IC), is now widely used to measure environmental specimens for eight anions. The IC technique is rapidly replacing the wet chemical methods of spectrophotometry, titrimetry, turbidimetry, and specific ions electrodes that are currently used.

A Dionex Model 16 ion chromatograph with AS-1 anion exchange separator columns was used for measuring these extracts. Direct injection of aqueous standards was used to establish a calibration curve; then direct injection of the aqueous extracts permitted comparison of anion peak heights with the standard curve to obtain concentrations in milligrams per liter.

The chromatograph utilizes a combination of ion exchange chromatography, eluent suppression, and conductimetric detection to measure low milligram-per-liter concentrations of F, Cl, Br, I, nitrate, nitrite, sulfate, and phosphate anions in aqueous specimens. A low-capacity anion exchange resin column separates the anions; then a suppressor column packed with high-capacity cation exchange resin converts the eluting solution ions (carbonate-bicarbonate) to carbonic acid. The carbonic acid electrical conductivity is very low, which permits the detection of 1 to 5 mg/L anion concentrations. The conductivity is easily related to the concentration of each anion present by measuring the conductivities of similar anions in external standard solutions.

The advantages of the ion chromatographic technique are rapid (30 min) sequential analysis of eight anions, minimal interference by other ions, and reduced or no sample preparation prior to analysis. Based on the analysis of internal controls and on the measurement of solutions prepared by the ORNL Analytical Chemistry Division Quality Assurance Officer, the precision of the analysis is ± 5% for fluoride, bromide, chloride, and iodide and ± 10% for sulfate, phosphate, nitrite, and nitrate. Figure 17 illustrates the separation of these anions.

RESULTS AND DISCUSSION

MUNICIPAL WASTE LEACHATE

Leachate from each of the two lysimeters (henceforth referred to as lysimeters A and B) was monitored relative to pH, Eh, and organic and inorganic constituents during the time in which MWL was used as a leaching medium (August 6 to October 20, 1982). Values for pH ranged from 3.5 to 6.0 (Fig. 18). Leachate from lysimeter B was slightly more acidic than leachate from lysimeter A. Values for Eh were also 50 to 100 mV lower for leachate from lysimeter B than A (Fig. 19). The difference is attributed to a small leakage of air into the sump recycling line (at a connector linking the PTFE tubing to the silicon peristaltic pump tubing) of lysimeter A. The leak was detected and stopped on August 21, 1982 (d 15 of leaching). On exposure of MWL to air, the pH and Eh become elevated, and a black precipitate coated the recycle line between the sump and glass manifold of lysimeter A. Such a phenomenon is well documented in the literature (Fuller et al. 1979). The erratic increase in pH and corresponding decrease in Eh (d 54 to 56) were caused by instrument malfunction.

Differences in Municipal Waste Leachate Over Time

Total organic carbon (TOC) content of the MWL decreased from approximately 3000 mg/L at the beginning of the leaching period to 1600 to 2000 mg/L at the end of the experiment. Leachate from lysimeter B appears to have contained slightly higher TOC concentrations than leachate from lysimeter A (Fig. 20) over the 79 d. Carboxylic acid concentrations in MWL from the two lysimeters appear to have followed a similar trend (Table 14) and make up about 72% of the TOC over the 79 d (Table 15). The fraction of TOC attributed to the carboxylic acids ranged from a high of 83% (observed after the first 7 d) to a low of 59% (measured about midway through the experiment at 48 d). However, there appeared to be a decreasing trend in the fraction of TOC made of carboxylic acids

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over the 79 d (Table 15). Analyses of leachates from lysimeters A and B, 30 d before leaching of the industrial wastes, revealed that carboxylic acid concentrations were considerably higher than during the leaching experiment, but were intermediate to those concentrations observed in MWL collected from the U.S. Corps of Engineer Waterways Experimental Station (WES) at Vicksburg, MS. Those leachates were collected at WES and analyzed at ORNL (Table 16). The MWL from WES (WES-I and WES-II) represented two different ages of leachate, 1.5 and 4.7 years, respectively, for WES-I and WES-II. Note the elevated ratios of the larger molecular weight carboxylic acids (concentrations of pentanoic and hexanoic acids relative to acetic acid) in the WES leachates as compared to those observed in the ORNL/MWL. The older WES/MWL (WES-II) also showed higher ratios of larger molecular weight carboxylic acids as compared to the younger leachates. This probably represents a decomposition and/or slower generation rate of acetic acid with age of MWL. Concentration of carboxylic acids in MWL will depend on a number of factors other than age (e.g., the character of the waste [percent organic content, ease of degradation], content and availability of essential nutrients required by an active microbial population, presence or absence of toxic metals, and rate of infiltrating water).

Concentrations of inorganic cations and anions in lysimeter leachate also decreased with time (Table 17). Note that the concentration of Na decreased from 600 mg/L, 30 d before leaching of the industrial wastes, to 115 mg/L at the end of the leaching. The changes, however, during leaching of the industrial wastes were not major (Table 17).

Differences in the concentration of TOC and the carboxylic acids over the leaching time (79 d) and potential differences between lysimeters A and B were tested using an analysis of covariance (day being the covariable). These analyses revealed that concentrations of TOC during the first 2 weeks were significantly higher ($P < 0.05$) than those measured in the last 3 weeks of leaching. However, concentrations in TOC after d 20 did not change significantly ($P < 0.05$) over the rest of the 79 d. Leachates from lysimeter A were also not significantly different than leachates from lysimeter B in regard to concentration of TOC. There was, however, a statistically ($P < 0.05$) higher concentration of acetic acid in leachates from lysimeter A (mean concentration: 1104 mg/L) than from lysimeter B (mean concentration: 988 mg/L). No differences between lysimeters were observed for concentrations of any of the other carboxylic acids. However, the concentrations of acetic, propionic, hexanoic, and octanoic acid content in both lysimeters varied significantly ($P < 0.05$) over the 79 d of leaching. On the other hand, concentrations of butyric and pentanoic acids remained relatively constant over time.

Concentrations of inorganic elements in MWL generated by lysimeter A or B appeared to differ little (Tables 18 and 19). The major difference was the higher concentration of Zn in MWL generated in lysimeter A as compared with lysimeter B.

Comparison of Oak Ridge National Laboratory Lysimeter Leachates

Many lysimeter research projects in recent years have investigated the physicochemical and leaching characteristics of MWL. One of the earliest and possibly largest and longest running is the one initiated in 1974 (Kinman et al. 1980, 1982; Strang 1977; Swartzbaugh et al. 1978; Walsh et al. 1979, 1981a,b,c). The purpose of this project was to observe the potential effects on the landfill environment of co-disposing industrial wastes with municipal solid wastes. Seven of the nine test cells (the dimensions of the test cells were identical to those of the ORNL study, 1.8 m in diameter by 3.6 m in height) were loaded with seven industrial wastes (sewage sludge, petroleum sludge, battery waste, electroplating waste, inorganic pigment waste, chlorine production brine sludge, and solvent-based paint sludge). The remaining two test cells were loaded with only municipal refuse for comparison purposes. These cells have been in operation since 1974. Most of the data on this project have been presented in terms of the fraction of material leached from the test cells relative to that originally placed in the cell (e.g., total milligrams of the element leached per kilogram of solid waste plotted against the moisture leached from the test cell) rather than in terms of the concentration of the constituent over time. Therefore, it is difficult to make comparisons of the leachate characteristics to the leachate generated in this study. Apparently, the pH of the leachate (the pH of leachate from test cell no. 2, which contained only municipal refuse, ranged between pH 6 and 7 over 2500 d of leaching; Fig. 4 in Kinman et al. 1982) is similar to the pH of the leachate generated in this study. The TOC in the leachate (4000 to 5000 mg/L) from cell no. 2, however, was considerably higher than TOC levels in this study (1400 to 3200 mg/L). The dates when these data were collected are not given, but they are assumed to be in 1977 or 1976; the data were reported in Swartzbaugh et al. (1978). Water was applied to simulate annual infiltration rates of 200, 400, 600, and 800 mm. Water was not applied uniformly; rather, it was applied at differential rates in accordance to the season (e.g., high rates were applied in the normally rainy months of March, April, and May). These annual infiltration rates corresponded to 0.39 to 1.54 $mL/min/m^2$, which is 4.5 to 17 times slower than the flux of water added in this study. The same group (Walsh et al. 1981c) has started another landfill simulation experiment, but the purpose of the new experiment is to demonstrate the potential for gas recovery and utilization at landfills. The objective of their new project is to test four techniques for the enhancement of gas generation; therefore, the TOC and volatile acids, measured as acetic (approximately 13,000 and 21,000 mg/L), were much higher than those in this study and comparison of the two studies is not valid.

Another long-term (5-year) comprehensive study, that involved the simulation of landfill MWL using lysimeters, was conducted by Fungaroli and Steiner (1979). Here the lysimeter contained approximately 1.8 Mg of waste, and water was added to it to simulate water infiltration in southeastern Pennsylvania. Annual flow rate was equivalent to 0.91 mL/min/m^2 (about 8 times faster than this study); however, no water was added during the summer months of June and July, but in January, February, and March, about 50% of the water was added (flux equivalent to 1.9 mL/min/m^2). The pH of the leachate ranged from 5 to 7 and TOC measurements were not taken; therefore, comparisons to this study cannot be made. Sodium concentrations averaged between 500 and 1000 mg/L and maximum values for Fe and Cl were 1600 and 2400 mg/L, respectively. Because of the variable inflow, leachate generation varied according to season. The system did not reach field capacity relative to water-holding capacity until approximately 500 d from the time water flow was started. The peak chemical oxygen demand (COD) was approximately 50,000 mg/L early in leachate generation (less than 50 d from the start of flow); however, the average COD during the first 2 years was 20,000 to 30,000 mg/L and was less than 2000 mg/L toward the end of the fifth year. The same researchers were monitoring a field cell in a landfill so that comparisons between the lysimeter and field site could be made. The overall objective of the research appeared to be the development of a computer model to simulate leachate movement from landfills. The researchers observed that concentrations of the constituents in lysimeter MWL were considerably higher than those observed in landfill MWL.

The data in Tables 20 and 21 have been tabulated to show the comparison of the chemical characteristics of the leachate generated by the lysimeters in this study to those leachates generated in other lysimeter experiments and to natural leachates sampled at municipal waste landfills. There appears to be little difference between the pH of the ORNL leachates and other leachates generated in lysimeters. If there is any difference, it would be that the ORNL leachates appear to be slightly more acidic. The anoxic environment in which the pH measurements were made might be responsible for this possible difference. The comparison between ORNL leachates and those produced in studies by Qasin and Burchinal (1976) and Meyers et al. (1980) indicate that the ORNL leachate is not as concentrated in terms of TOC, Ca, K, etc.

In the case of the Qasin and Burchinal (1976) study, the "metals, cans, bottles, stones, and large pieces of wood" were removed from the municipal waste (from Morgantown, West Virginia) before the waste was placed in the 0.9-m diameter lysimeters (about one-half the size of the ORNL lysimeters, although cylinder heights were 1.2, 2.4, 3.6, and 4.8 m). The leachate was younger (20 to 160 d) than the ORNL leachate (160 to 240 d during the leaching of the industrial wastes), and water was added to the lysimeter (3.7 $mL/min/m^2$) at about one-half the flow rate of that added to the ORNL lysimeters.

Meyers et al. (1980) used the same size test cells as ORNL, but their flow rate of distilled water as influent was about 5.5 times slower (1.26 $mL/min/m^2$). The age of control leachates was 8 to 17 months, which makes them 2 to 3 times older than the ORNL leachate. These comparisons show that the ORNL leachate is generally a "weaker" MWL than the MWL generated by other lysimeter test facilities. The most likely reason for the lower concentrations of TOC and inorganic elemental constituents is the faster flow of water to the lysimeter. The ORNL flow rate was used so that sufficient leachate was generated to leach the industrial wastes (packed to simulate a 95:5 municipal/industrial waste scenario) to the desired 20:1 liquid/solid ratio over the experimental timetable of 80 to 100 d.

The ORNL leachate resembles more closely the characteristics of natural leachates collected from municipal waste landfills than leachates generated in lysimeter test facilities (Tables 20 and 21). The amount of data available on carboxylic acid content is quite limited. Note that the data set used by Chian and DeWalle (1977) represents four leachates selected to test the effect of carboxylic acids in MWL on the ability to analyze for the heavy metals Cu, Pb, Cd, Cr, and Ni by AAS; therefore, these likely represent a data set characteristically higher in carboxylic acids than the "average" MWL. The range in concentrations of the selected parameters, or characteristics, of the natural leachates from landfills is quite wide. The average TOC concentrations of the leachates analyzed by Chian and DeWalle (1977) and Johansen and Carlson (1976) was 582 mg/L, which is lower than the ORNL lysimeter leachate (the other studies in Table 20 reported ranges over the age of the leachate, making it impossible to calculate an average TOC value). As noted previously by Fungaroli and Steiner (1979), it appears that leachates from lysimeter test facilities characteristically are more concentrated with respect to both carboxylic acids and inorganic constituents than MWL measured under field conditions.

The wide range in MWL composition also results from dilution effects by water from outside sources. For example, for many landfills transitory breachments by surface runoff and/or shallow land groundwater result in major changes in leachate composition. These breachments are the major forces in contaminant dispersion from the landfill. Consequently, there is not such an entity as a "typical" MWL.

FIELD LYSIMETER STUDIES

Aggressor

After 28 d of leaching MWL through the four industrial wastes (0.8 kg per column), the liquid/solid ratio averaged 19.3 ± 3.8 (n = 20). The average pH of the leachates varied from 8.27 to 5.35 (Table 22). Because considerable fine particulate matter was present in some of the waste leachate, aliquots were analyzed for inorganics by ICP before and after filtration to evaluate whether there was a difference in concentrations of elemental inorganics between filtered and nonfiltered leachates. The purpose was to thoroughly assess total loss of the chemical constituents from the industrial wastes. From the appearance of some of the leachates, it was readily apparent that considerable particulate had formed as a result of entrainment by the MWL or by secondary precipitation on storage of the leachate. The probable cause of particulate formation was secondary precipitation on aging of the leachate, as fresh leachate did not visibly contain as much particulate as aged leachate.

Concentrations of Inorganic Elements in Leachates: Analyses of the leachates revealed elemental concentrations statistically different ($P < 0.05$) than those measured in the leachate of the control treatments (Table 23). Note that there were two controls: (1) a sand control (columns containing only sand, replicated twice) for comparison with the API/incinerator ash and electroplating waste treatments and (2) a sand-sawdust control (columns containing only sand and sawdust, replicated twice) for comparison with the dichloroethylene still bottoms and paint sludge. Of the inorganics targeted before analyses of the industrial waste leachates (see Chemical Analyses section); only one target inorganic, Cr in the API/incinerator ash leachate, was found at concentrations greater than 1 mg/L (Table 24). Also in Table 24, note the very low concentrations of the target inorganic elements (As, Cd, Cr, Pb, and Ni) in the control ORNL/MWL.

Leachates from the API/incinerator ash contained concentrations of Ca, Cr, K, Mg, Mo, Na, and Sr significantly higher ($P < 0.05$) than those observed in the control leachates (Table 23). Concentrations of Fe, Mn, P, and Zn were significantly lower than the concentrations in the control leachates. The low concentrations of these elements were caused by their relatively low mobility and solubility in the high pH waste/leachate matrix (average leachate pH of 8.3). Phosphorus levels were below ICP detection levels. Also, note that concentrations of Fe and Cr were higher in the nonfiltered leachates than in the filtered leachates (Table 25).

As expected, because of the inherently low concentrations of the inorganic constituents in the waste, elemental inorganic concentrations in the leachates of the dichloroethylene still bottoms appeared to be similar to those measured in the control leachates (Table 23). Also, concentrations of Fe and P were lower in filtered leachates than in nonfiltered leachates (Table 26).

Levels of Ba and Zn in the leachates of the paint sludge were observed to be statistically greater ($P < 0.05$) than their respective concentrations in the leachates of the sand-sawdust controls. Concentrations of Fe, Mo, P, and Sb were lower in filtered than nonfiltered leachates (Table 27). The fact that Fe and P concentrations in the filtered leachates of the paint sludge and the dichloroethylene still bottoms were lower than concentrations measured in the nonfiltered leachates indicates that factors other than pH may have influenced formation of secondary precipitation. The pH of the leachate obviously influences the total concentration of Fe and P in the leachate. Note the much lower concentrations in the API/incinerator ash leachate (which has a high pH, 8.3) compared to those in the still bottoms, paint sludge, electroplating waste, and control leachates (Table 23). However, even these latter leachates, which are mildly acidic (average pH ranged from 5.35 to 6.28; see Table 22), showed lower concentrations of Fe and P in the filtered samples than in the nonfiltered samples (Tables 25 to 28). This lower concentration indicates the formation of secondary precipitation products on exposure of the leachates to air. Most likely, what occurred was the oxidation of ferrous iron to ferric iron with subsequent precipitation of iron sesquioxides; these in turn coprecipitated P, Cr, and possibly Mo and Sb from the leachates. However, concentrations of the other inorganic elements were not noticeably affected.

Leachates from the electroplating waste contained concentrations of B, Ba, K, Mn, Mo, Na, Ni, P, Sr, and Zn that were significantly higher ($P < 0.05$) than those observed in the control leachates (Table 23). Concentrations of Fe, like those in the leachate from the API/incinerator ash, were statistically lower ($P < 0.05$) than the control leachate concentrations. However, unlike the API/incinerator ash leachate, the concentrations of Mn and P in the electroplating waste leachates were higher than the control leachates. This difference is probably a result of the much lower average pH (6.28) of the electroplating leachate relative to the average pH (8.27) of the API/incinerator ash leachates.

In summary, the major contaminating inorganic constituents observed in leachates from the four industrial wastes appear to be: (1) Cr and Mo in the API/incinerator ash, (2) Ba and Zn in the paint sludge, and (3) Ba, B, Mo, Ni, and Zn in the electroplating waste. Of course, the generally high concentrations of Na, K, and, to a certain extent, Ca in leachates from these industrial wastes would appreciably lower the water quality of surface and groundwaters with which they come in contact.

Fraction of Inorganic Elements Leached: An estimate of the fraction of the inorganic constituents leached from the industrial wastes by the MWL over the 28 d can be determined using the data in Tables 1, 22, and 23. The results of these calculations for the elements whose concentrations in the industrial waste leachate were significantly greater than those measured in the control leachates are presented in Table 29. The concentrations of the elements added by the MWL were calculated using control leachate concentrations and the liquid/solid ratio for that waste leachate multiplied by 0.8 kg to determine the liters of leachate collected and subtracted from those concentrations measured in the industrial waste leachate. The percentage extracted varied from a low of 0.13% (for Ba in the paint sludge) to fractions in excess of 100% (Na from API/incinerator ash and electroplating waste). The reason for the high value for Na is unknown. One likely source of error is that the relatively small sample size taken for total analyses (0.100 g) was not a representative sample of these wastes.

Concentrations of Organic Compounds in Leachates: Analyses of the leachates revealed progressively higher concentrations of the target compounds in API/incinerator ash, dichloroethylene still bottoms, and paint sludge leachates (Table 30). These differences are similar to the relative difference in the levels of these compounds found in the wastes (Table 2). The concentration of naphthalene in API/incinerator ash leachates, although low compared to ethoxyethanol and ethoxyethyl acetate in the paint sludge leachate, is high considering the relatively low solubility of naphthalene and the concentration in the original waste. The percentage leached was 11.5% (Table 31). On the other hand, only a small proportion (about 0.04%) of the HCB was shown to leach. This is surprising, not so much from the standpoint of solubility, but because of the very high amount of this compound originally present (i.e., 22.4%). It would be tempting to suggest that some channeling might have reduced contact between the wastes and leaching medium except that the other components of the waste were leached at considerably higher levels. Dichloroethane and trichloroethane were leached at similar levels (58 and 56%, respectively). As was the case throughout this research, precision for the volatile halocarbons was poorer than for any of the other organic target compounds. This was in part due to the high volatility of these compounds and potential differential losses and in part due to the occasional presence of interferences in the control MWL. With respect to the paint sludge, 14% of the xylenes was leached. This rate is similar to that of naphthalene. Although the physical properties of the two wastes were dissimilar and the levels of naphthalene and xylene were grossly different, the similar leaching rates indicate that the solubility in MWL is a prime factor in the mobility of organic compounds. All of the highly soluble ethoxyethanol and moderately soluble toluene (123 and 130% of the estimated waste content, respectively) was leached from the paint sludge. With respect to the ethoxyethyl acetate, approximately 1.6 times more was detected in the leachate than expected based on measurement of total content in the waste. It is not clear whether this discrepancy was a result of an experimental error (sample collection, storage, etc.), analytical error (dissolution and detection errors), or possible interference from MWL. Although no such interference was detected, the possibility cannot be ruled out.

Comparisons between Concentrations in Aggressor Leachates and EP Extracts: The data available from Aggressor provide an excellent opportunity to assess the ability of EP to simulate the leaching of industrial wastes in a co-disposal environment. Some comparisons between the concentrations of inorganic and organic constituents extracted from the wastes by the EP and those observed in the leachates from Aggressor are presented in Tables 32 to 35. The inorganic constituents listed in these tables are those elements in the industrial waste leachates of Aggressor that were measured in statistically higher concentrations ($P < 0.05$) than those measured in the control leachates. Concentrations in industrial waste leachate minus the applicable control leachate are used throughout Tables 32 to 35 to reflect the leaching capacity of MWL.

Many of the NIPDWS elemental concentrations measured in both the EP extracts and Aggressor leachates were very low, reflecting background concentrations, and comparison between the two has little meaning. The arbitrary criterion for a "match" between the EP and Aggressor concentrations is that the EP concentration should be less than 1.5, but more than 0.5, times the Aggressor concentration (ratios of EP/Aggressor between 1.5 and 0.5:1). Using this criterion the following can be stated:

1. For the API/incinerator ash, the EP overestimated Ca, Cr, Mg, and Sr, underestimated Na and K, but was a good estimator for Mo and naphthalene;
2. For dichloroethylene still bottoms, the EP overestimated HCB but underestimated dichloroethylene, trichloroethylene, dichloroethane, and trichloroethane.
3. For paint sludge, the EP overestimated Ba and slightly underestimated toluene and the xylenes, but was a good estimator for ethoxyethanol, ethoxyethyl acetate, and Zn; and

4. For electroplating waste, the EP overestimated Cr, Mn, K, P, and Zn, but was a good estimator for B, Ni, and Na.

Other criteria for comparison of concentrations between the EP extracts and Aggressor leachates may be equally valid. If only the potentially toxic constituents (i.e., Cr, Mo, and naphthalene) in the API/incinerator ash; the organic compounds in the dichloroethylene still bottoms; Ba and Zn in the paint sludge; and B, Ni, and Zn in the electroplating waste were considered, then major differences in concentrations between the EP and Aggressor leachates occurred only in the cases of Cr in the API/incinerator ash and the organic compounds in dichloroethylene still bottoms.

In general, the EP tended to overestimate acid-soluble inorganics in alkaline base wastes (Cr in API/incinerator ash and Zn in electroplating waste). Note that the pH of EP extracts is considerably lower than the pH of the Aggressor leachates for these wastes (Tables 32 and 35). The lower concentration of Cr in MWL probably results from the reduction of Cr(VI) to Cr(III) by MWL. Chromium (III) is an oxidation state much less mobile than the Cr(VI) form present in the API/incinerator ash. The acetic acid and oxic environment of the EP do not cause a similar reduction of Cr(VI); consequently, the EP extracted measurably more Cr than the MWL. Total Cr concentrations in the electroplating waste (16,500 mg/kg) were considerably higher than those measured in the API/incinerator ash (3900 mg/kg, Table 1); however, more Cr was extracted by both the EP and the Aggressor MWL from the API/incinerator ash than from the electroplating waste, suggesting that the Cr in the electroplating waste was in a reduced Cr(III) form or some other very insoluble form. The EP, however, simulated quite well the concentration of Ni extracted from the electroplating waste by MWL (Table 35).

Target

The objective of this experiment was to determine the concentrations of inorganic and organic constituents in waste leachates over a leaching interval equivalent to a 20:1 liquid/solid ratio. The purpose was to identify leachable constituents and characterize their leaching pattern in terms of concentration and time. The intent was to target inorganic and organic constituents and determine their concentration over time so that a laboratory leaching method could be developed to simulate selected segments of that leaching pattern.

Volumes of leachate collected from the 16 waste columns averaged slightly less than the intended 20:1 liquid/solid ratio (Table 36). Over the 79 d of operation, the average flow rate (0.6 mL/min) to each column was approximately 25% slower than the tubing specifications (0.8 mL/min); however, the low coefficient of variability (approximately 10%) among the 16 columns was considerably better than anticipated.

Leachate was sampled twice a week during the first month of operation and once a week for the remainder of the experiment. Inorganic (ICP) analyses indicated that leachates from the industrial wastes contained several elements that showed concentrations in excess of those observed in the leachates from the sand and sand-sawdust controls (Figs. 21 through 26, 32, 33, and 38 through 45). The inorganic elemental concentrations shown in these figures are averages calculated from filtered and unfiltered leachates. The elements include:

1. Ca, Cr, K, Na, Mo, and Sr from the API/incinerator ash;
2. Ba and Zn from the paint sludge; and
3. B, Ba, K, Mn, Na, Ni, Sr, and Zn from the electroplating waste.

Many of these elements are not typically considered to be toxic (e.g., Ca, Na, K, Mg, and Sr). However, they do represent matrix elements of the waste, and their dissolution and leaching by MWL are indicators of weathering of the wastes in a co-disposal environment. A laboratory extraction procedure that simulates their leachate concentration is useful because it predicts the stability of that waste in a co-disposal environment, and for that reason all of the above listed elements will be considered targets for the comparison of laboratory extraction methods.

Organic compounds found to be leached from the industrial wastes in excess of the leaching observed in control MWL (Figs. 27 through 31 and 34 through 37) over the 79 d of leaching were:

1. Naphthalene from the API/incinerator ash;
2. Dichloroethane, trichloroethane, trichloroethylene, and HCB from the dichloroethylene still bottoms; and
3. Ethoxyethanol, ethoxyethyl acetate, toluene, and xylenes from the paint sludge.

These organic compounds and the inorganic elements listed above constitute the list of targets that are used to make comparisons among extraction methods to best simulate lysimeter leachate concentrations.

Target Concentrations: Target concentrations are the concentrations of the organic and inorganic target constituents observed in the lysimeter leachates that the EP-III will model. The concentrations of organic and inorganic target constituents in each of the laboratory extractions will be compared to the lysimeter leachate target concentrations, and the method or methods that most closely approximates the lysimeter leachate target concentrations will form EP-III. The criteria used to establish the target concentrations in the lysimeter leachates depend on the intended use of the solid waste extraction procedure. Several alternatives exist:

1. Maximum concentration observed in lysimeter leachate over 79 d;
2. The average concentration integrated over some predetermined leaching interval bracketing (or

containing) the maximum observed leachate concentration; and

3. The average concentration integrated over some predetermined leaching interval beginning the first day of leaching irrespective of the maximum concentration.

The first alternative would depict the most aggressive and the most conservative extraction method with respect to classifying a waste as potentially toxic (i.e., it would tend to extract higher concentrations of toxic constituents [and thus classify more wastes as toxic] than extraction methods based on either of the other two alternatives). The concern in adopting such a criterion is that for most wastes the maximum leaching concentration emanating from a landfill will be short-lived in a temporal and spatial sense (see Figs. 21 through 45). Thus, this first alternative would produce an extraction method that represents landfill leachate concentrations over a very short leaching interval.

The second alternative is a modification of the first in that an average concentration is determined over a leaching interval that encompasses the period of time (in terms of liquid/solid ratios) during which the maximum concentration in the lysimeter leachate was observed. An extraction method that accurately models target values obtained using this alternative would be less aggressive than the first alternative and consequently more moderate with respect to identifying wastes likely to contaminate subsurface environments adjacent to a landfill. The primary issue with this alternative is selection of the appropriate leaching interval. The intent is to select a leaching interval that encompasses the maximum concentration at its midpoint. As an example, the leaching curve for zinc in the electroplating waste (Fig. 45) shows a maximum leachate concentration occurring at an average liquid/solid ratio of 14:1. If a liquid/solid ratio of 8:1 were assumed to be an appropriate leaching interval, the target concentration based on the second alternative would involve determining the average zinc concentration between a 10:1 and 18:1

liquid/solid ratio. The important issue here, as with the third alternative, is selecting the appropriate leaching interval.

The third alternative involves integrating the concentration of a constituent as it is leached from a waste over a selected leaching interval beginning d 1. It basically represents the total quantity of a constituent leached from a waste over a selected leaching time, divided by the volume of leachate. This differs from the second alternative in that it is not based on maximum leachate concentrations. Because peak concentrations from the lysimeter would be integrated over background concentrations (those concentrations similar to the controls), the third alternative would reflect the least aggressive extraction method and be the least conservative of the three alternatives. For example, for those wastes with readily soluble constituents, the longer the leaching interval over which the target concentration is integrated, the lower the resulting target value will be. On the other hand, for those wastes whose dissolution of the target constituents increases upon continued interaction with the MWL, the target concentration will increase over the longer interval. For wastes whose target constituents are limited by solubility in the MWL, the target concentration will be independent of the leaching interval selected. This alternative would result in an extraction method that predicts average leachate concentrations over a leaching interval starting from d 1. As such, it will not predict the higher concentrations of constituents that could result from continued leaching of the waste.

For example, using the leaching profile of zinc from the electroplating wastes again (Fig. 45), and assuming again that a liquid/solid ratio of 8:1 is an appropriate leaching interval, the target concentration based on the third alternative would simply be the average concentration over the first 8:1 liquid/solid ratio. The extraction method based on this alternative would not predict the higher concentrations of zinc observed on subsequent leaching intervals (concentrations on the order of 150 mg/L at liquid/solid ratios greater than 10:1; see Fig. 45).

The primary issue in the use of the latter two alternatives is the selection of the appropriate leaching interval. For example, the quantity of leachate (generated from groundwater that infiltrates the site or rain water that percolates through the landfill) that moves through a landfill is dependent on numerous geologic, meteorologic, and hydrologic factors as well as the engineering methodology used in filling and closing the landfill. Ideally the selection of the leaching interval should approximate or reflect these factors in modeling the movement of the leachate.

The intent of this research is to establish target concentrations using all three alternatives and then to rank the various laboratory extraction methods depending on which method most closely simulates the particular target concentration. Five sets of target concentrations for the 25 inorganic and organic target constituents have been established based on the guidelines developed by Kimmell and Friedman (draft manuscript entitled "Models Assumptions and Rationale Behind the Development of EP-III" to be presented at the Fourth Symposium for Hazardous and Industrial Solid Waste Testing, May 2-4, 1984, Arlington, Virginia). The first set was determined using the criterion of alternative one. Three of the five sets were established using the criterion developed for the second alternative over three different liquid/solid ratios. The fifth set of target concentrations was established using the integrated average concentration (third alternative) over an 8:1 liquid/solid ratio. The five sets are as follows:

1. MLC - maximum leachate concentration measured in lysimeter leachates over 79 d leaching,
2. AMC8 - average maximum concentration in an 8:1 liquid/solid leaching interval that brackets the maximum leachate concentration measured in lysimeter leachate,

3. AMC20 - average maximum concentration in a 20:1 liquid/solid leaching interval that brackets the maximum leachate concentration measured in lysimeter leachate,
4. AMC40 - average maximum concentration in a 40:1 liquid/solid leaching interval that brackets the maximum leachate concentration measured in lysimeter leachate, and
5. IAC8 - integrated average concentration leached over the first 8:1 liquid/solid ratio.

The 8:1 liquid/solid ratio was selected because the preliminary assumptions relating to the quantity of leachate that moved through a municipal waste landfill relative to the industrial waste in a landfill indicated that an 8:1 liquid/solid ratio represented a period of leaching equivalent to 1 to 3 years, depending on the assumptions regarding meteorologic and geologic conditions and landfill design. The 20:1 liquid/solid ratio was used in that it would represent a significantly longer leaching period, and it is also the same liquid/solid ratio currently being used in the RCRA EP. The 40:1 ratio was included primarily to illustrate possible long-term leaching characteristics of the various target constituents.

To determine MLC target values, an attempt was made to use a linear fit for the initial portion of the curves that represented rapid leaching of water-soluble constituents (e.g., B in the electroplating waste, Zn in the paint sludge, and many of the organic compounds in the dichloroethylene still bottoms and paint sludge). The intent was to fit these curves over the initial part of the leaching curve and to use the intercept of this curve with the first pore volume (the quantity of water held at field capacity expressed as a fraction of the total volume occupied by the waste) to determine MLC values for the various constituents. This approach was recommended by a peer review team established to provide technical assistance and guidance to the project. The review team consisted of M. A. Anderson (University of Wisconsin, Madison, Wisconsin), D.R. Brunner (E. J. Jordon Co., Portland, Maine), and C. S. Helling (U.S. Agricultural Research Service, Beltsville, Maryland). The problem, however, was deciding on an acceptable rationale for terminating the linear fit (i.e., should the linear fit be used for liquid/solid ratios covering the range of 0 to 5, 0 to 7.5, or 0 to 10?). If this approach were to be used, rationale about the range of liquid/solid ratios that was consistent and acceptable in fitting data for the variety of leaching profiles across all target/waste combinations would have to be established. Such a rationale was not found to be practical. Thus, in keeping with the objective of the MLC criteria, the values for MLC were derived by taking the average maximum concentration measured over the 79 d in the four replicated industrial waste leachates. Control concentrations for each replicate were subtracted at the MLC solid/liquid ratio. This rationale is consistent for all leaching curves, and it provides what was sought--the maximum concentration of that constituent in the industrial waste leachate. These concentrations, and their respective lower and upper 95% confidence levels, are presented in Table 37. Also presented in this table are the mean liquid/solid ratios for MLC target concentrations.

Target concentrations for AMC8, AMC20, AMC40, and IAC8 were determined as follows. First, the accumulative leaching curves for each of the 25 target constituents were fitted using the best fit curve from a selection of four basic single variable models (Daniel and Wood 1980):

1. $Y = a + bX$,
2. $Y = ae^{1/X}$
3. $Y = ae^{bX}$, and
4. $Y = a\ln X$,

where Y = total quantity of a constituent leached (in milligrams); X = liquid/solid ratio; and a and b are constants for a particular curve. Fitting of the accumulative leaching curves to one of these four models provided equations so that quantities of the leached constituents could be calculated over discrete liquid/solid ratios. It also provided capabilities to extrapolate to larger liquid/solid ratios (e.g., 40:1)—however, extrapolations of this nature should be viewed with extreme caution.

The fitted accumulative leaching curves and the respective leaching data for each of the 25 target constituents are presented in Figs. 48 to 72. Using these fitted leaching curves, the quantity of each target constituent was calculated for selected liquid/solid ratios. AMC target concentrations were calculated for liquid/solid ratios of 8, 20, and 40:1, bracketing (or containing) the MLC for that replicate. For example, to calculate the AMC8, the quantity of a target constituent leached was determined over a liquid/solid ratio of 4:1 on each side of the MLC liquid/solid ratio. After subtracting the quantity leached in the appropriate control over the same liquid/solid ratio, this quantity (in milligrams) was then converted to an average concentration over that liquid/solid ratio by dividing by the volume of leachate (in liters) collected during that interval. If the MLC liquid/solid ratio was less than 4, then the quantity leached over the initial liquid/solid ratio of 8 was used. The same methodology was used to calculate AMC20 and AMC40 using liquid/solid ratios of 20 and 40, respectively, as the intervals over which the average concentration is determined. Figure 73 illustrates this concept.

To calculate IAC8, the total quantity of the target constituent leached over the first eight liquid/solid ratios was determined followed by subtracting the quantity leached in the control at the same liquid/solid ratio and dividing the difference by the volume of leachate collected.

Target concentrations for the 25 inorganic and organic constituents in terms of MLC, AMC8, AMC20, AMC40, and IAC8 are listed in Tables 37 to 41 (a summary is presented in Table 61).

Leaching of Inorganic Elements: Numerous factors contribute to the leaching pattern of the various inorganic elements present in the waste leachates. Leaching is typified by two basic leaching curves: (1) a curve that depicts the rapid leaching of readily water-soluble constituents from the waste (such as B and Zn from the paint sludge), and (2) a curve in which the initial release is low with increasingly higher concentrations being leached with time, such as Zn and Ni from the electroplating waste. For the case of the relatively water-soluble constituents (e.g., Cr, K, Na, and Mo in the API/incinerator ash; Ba and Zn in the paint sludge; and B, Na, and K in the electroplating waste), the maximum leachate concentration (MLC) will depend on the leaching rate of the fresh influent. For these elements, water-solubility is not greatly limited by the pH of the waste/MWL interactions and high infiltration rates of the MWL will result in lower MLC concentrations than slow infiltration rates for a given quantity of waste. However, for such elements as Ni and Zn in the electroplating waste, and (to a certain extent) Ca, Mg, and Sr in either the API/incinerator ash or electroplating waste, the leaching profile is highly dependent on the chemical characteristics of the leaching medium and on how rapidly the leaching medium (MWL) dominates the environmental chemistry of the system. For example, as MWL interacts with the electroplating waste, the pH of the leachate decreases from approximately 8.5 to near 6.0 at the end of the 79 d (Fig. 74). As the pH is lowered, the solubility of Ni and Zn is increased, resulting in maximum concentrations of these elements at liquid/solid ratios of 7.6 and 14.0.

Fraction of Inorganic Elements Leached: Less than 1% of the Ba in the paint sludge, was leached by the MWL over the 79 d (Table 42), an amount similar to that leached in the 28 d leaching for Aggressor (Table 29). In fact, the fraction of all inorganic elements leached in Target is quite similar to that leached in Aggressor. The one notable exception is the higher percentage of Zn leached from the electroplating waste in Target (74%) as compared to that leached in Aggressor (9.7%). The basic reason for this difference is the increasing solubility of Zn as the pH of the leachate becomes acid (Fig. 74). The final pH of the leachate is not as important as the time over which the waste was exposed to the acid leachate. For example, Fig. 74 shows that the pH of the electroplating waste leachate leveled off between 6.5 and 6.2 after a liquid/solid ratio of approximately 7. This means that in Target the waste was leached for about 50 d at a pH less than 6.5, while in the case of Aggressor the waste was exposed to a similar pH for less than 20 d. As the pH of the leachate becomes more acid, Ni is leached even more rapidly than Zn (see Figs. 43 and 45 and Table 37, which shows that the MLC liquid/solid ratio for Ni is 7.6 as compared to 14 for Zn). This accounts for the similar fractions of Ni leached in Aggressor and Target.

Leaching of Organic Compounds: As stated earlier, the objective of the lysimeter experiment Target was to define the time course of toxicant mobility in general and to define target concentrations of the toxicants using the previously mentioned criteria. Because all of the target organic compounds are neutral compounds, it would be reasonable to assume that their mobilities would be governed primarily by the liquid/solid ratio. The mass of a compound found in the MWL would, therefore, depend on the solubility of the component in MWL and the interactive forces between the component and the waste matrix. The interactive forces between the component and the waste would be expected to be relatively constant throughout the experiment. Solubility could be affected by changes in the chemical composition of MWL; these changes have already been shown to be minimal for the organic compounds. Therefore, only two types of leaching curves could be defended on the basis of physical principles: (1) an exponential decay and (2) a plateau followed by an exponential decay. The first case would apply to organic compounds whose initial concentration does not exceed the solubility of that compound in MWL. The second case applies to compounds whose initial concentration exceeds the MWL solubility. The plateau would continue until the solubility is no longer exceeded.

Several possibilities exist for explanation of non-ideal behavior. Initial application of MWL to the columns might not have been completely uniform, resulting in unsaturated zones within the waste. Under these conditions, the resulting leaching curve would reach its maximum at a point slightly later than time = 0, thus giving the appearance of a "peak." The presence of erratic (non-constant) interference in MWL could cause a peak to be observed that does not result from leaching of the industrial waste. Physical displacement of the waste itself could also cause significant aberrations in the leaching curves, as could other routes of removal (e.g., evaporation).

The leaching curves for the target organic components are shown in Figs. 27 to 31 and 34 to 37. The curves for xylene and toluene from the API/incinerator ash are also shown (Figs. 46 and 47). The behavior of naphthalene indicates a slight delay relative to the beginning of the experiment before the peak was reached. The remainder of the experiment showed the expected exponential decay, which probably occurred because the surface of the waste was oily and therefore the equilibrium distribution was not rapidly attained. This explanation is given credence by the fact that toluene and xylene from the paint sludge (Figs. 36 and 37) leaching curves showed the same type of delay. The leaching curves for dichloroethane in the dichloroethylene still bottoms showed essentially a plateau (solubility limited) followed by a gradual decay (Fig. 28). The curves for trichloroethane and trichloroethylene behave as predicted, although some scatter is noted. These compounds tended to have some unexplainable interferences in their measurements in the ORNL/MWL, which accounted for some of the anomaly (these are the only components interfered with by MWL). Excluding the single value at > 10,000 mg/L, which was caused by the biphasic leachate sample from the dichloroethylene still bottoms, HCB showed an essentially flat leaching curve (Fig. 31) with a relatively constant concentration throughout the experiment. The 10,000 mg/L value was apparently sampled from a nonaqueous phase in the dichloroethylene still bottoms leachate, as initial leachates collected from this waste showed a tendency to produce nonaqueous and aqueous phases. This resulted largely because of the pressure applied to the sawdust admixture as a consequence of layering sand on top of the admixture. None of the other wastes exhibited biphasic leachates. Similar effects may also occur in "real world" landfills; thus, the data point was used as sampled. It is possible that this compound was solubility-limited in its extractability from aqueous phases. The four components of the paint sludge (ethoxyethanol, ethoxyethyl acetate, toluene, and xylenes) all behaved as expected. In general, the solubility of the individual components affects the slope of the leaching curve; the components of highest solubility resulting in the steepest leaching curve.

Fraction of Organic Compounds Leached: In Target, as in Aggressor, the fraction of HCB leached from the API/incinerator ash was very small, less than 1% (Table 43). In fact, the fraction of target organic compounds leached in Target was quite similar to that leached in Aggressor. Two major exceptions were the lower fractions of trichloroethylene (41.8 vs 128%) and toluene (6.3 vs 130%) leached in Target as compared to Aggressor (Table 31). As in Aggressor, the difficulties in accurately determining concentrations of the more volatile organic compounds in the leachates as well as in the bulk sample contributed to the inability to make a satisfactory mass balance on these compounds.

Leaching Differences Between Lysimeters: The potential differences in the leaching ability of MWL from the two lysimeters, A and B, were tested using an analysis of covariance (day being the covariable). Except for the leaching of tricloroethane from the dichloroethylene still bottoms, there were no significant differences ($P < 0.05$) between lysimeters to leach target constituents from their respective wastes. The average concentration of trichlorethane leached from the dichloroethylene still bottoms by lysimeter A (52.8 mg/L) was higher than that leached by lysimeter B (40.4 mg/L). There was no other evidence that the two lysimeters produced a leachate that differed in its ability to leach target constituents, even though earlier analyses had shown significant differences in carboxylic acid content and Zn concentrations (see Results and Discussion section, "Municipal Waste Leachates").

LABORATORY EXTRACTIONS

Extractions on each of the four wastes were conducted using two procedures (upflow-column and rotary-batch) and four media: (1) 0.1 M sodium acetate pH 5, (2) a weak carbonic acid solution made by saturating de-ionized distilled water with carbon dioxide; (3) de-ionized distilled water; and (4) MWL from the field lysimeter test facility (ORNL/MWL). Four liquid/solid ratios were used: 2.5, 5, 10, and 20:1. The extracts were analyzed for the 25 target inorganic and organic constituents. With respect to the 0.1 M sodium acetate and ORNL/MWL media, control concentrations for Na, Ca, K, and Mn were subtracted. Comparisons between the rotary-batch and upflow-column procedures across these media are illustrated in Figs. 75 to 99. (Note in these figures the solid and dashed lines connect average concentrations calculated from the two replicates shown as stars and squares for batch and column procedures, respectively.)

API/Incinerator Ash

Calcium was extracted at higher concentrations from the API/incinerator ash by upflow-column extraction procedures using all media (except 0.1 M sodium acetate) than by rotary-batch procedures (Fig. 75). For example, across all extracting media and liquid/solid ratios, the upflow-column procedure extracted significantly more ($P < 0.05$) Ca than the rotary-batch procedure (Table 44). The ORNL/MWL extractions contained the highest Ca concentrations (averaging 929 mg/L, which was significantly greater [$P < 0.05$] than all other media). The acetate extractions contained the next highest (814 mg/L), which were higher ($P < 0.05$) than those for either the carbonic acid or the de-ionized water extractions. Across all media and extraction procedures, no significant differences were observed in the average concentration of Ca across the various solid/liquid ratios.

At liquid/solid ratios greater than 2.5:1, the use of carbonic acid or de-ionized distilled water as extracting media revealed little difference in the concentration of Cr extracted (Fig. 76). The column extraction procedure appeared to extract more Cr using ORNL/MWL as the extracting medium, whereas the rotary-batch procedure extracted more using acetate pH 5 buffer as an extracting medium, especially at the 2.5 liquid/solid ratio (130 as compared to 82 mg/L by column). Overall, there was not a statistically significant difference ($P < 0.05$) between concentrations of Cr measured in batch or column extracts (Table 44). Of the four media, de-ionized distilled water extracted the least (51.3 mg/L) and carbon-dioxide-saturated water the most (68.6 mg/L), across the two procedures and all liquid/solid ratios (Table 44). Possibly the most important point is that all media, regardless of procedure (upflow-column or rotary-batch) when used at liquid/solid ratios greater than 2.5, resulted in extracts containing Cr concentrations within the upper and lower 95% confidence levels for MLC target concentrations (25 to 101 mg/L; see Table 37). At 2.5 liquid/solid ratios, higher average concentrations of Cr were obtained in many of the extracts than those observed in the field lysimeter leachates (compare Fig. 22, field leachates, with Fig. 76, laboratory extracts).

The extraction of Mo from the API/incinerator ash appeared to be independent of the four media used (Fig. 77). However, statistical analysis revealed that ORNL/MWL and acetate buffer extracted significantly ($P < 0.05$) higher concentrations than de-ionized distilled water (Table 44). The rotary-batch procedure, using 0.1 $\underline{M}$ acetate as extracting media, appeared to extract slightly more Mo than column extractions (Fig. 77). The rotary-batch at a liquid/solid ratio of 2.5 using carbonic acid also appeared to extract more Mo, but otherwise little difference in the extraction of Mo was observed using rotary-batch or upflow-column procedures. Overall, the rotary-batch extracts had higher concentrations ($P < 0.05$) than did the upflow-column extracts (Table 44). Like Cr extractions, significantly higher concentrations of Mo were observed at each decreasing liquid/solid ratio.

Other than the ORNL/MWL extractions, K appeared not to be preferentially extracted by either upflow-column or rotary-batch procedures (Fig. 78). With respect to the ORNL/MWL, considerably more K was observed in rotary-batch extractions as compared to the upflow-column extractions. Also, the rotary-batch extractions contained nearly twice as much K as the upflow-column extractions using 0.1 M sodium acetate at a 2.5:1 liquid/solid ratio. Thus, across all media and liquid/solid ratios, the rotary-batch procedure extracted more ($P < 0.05$) K than the upflow-column procedure (averaging respective concentrations of 340 and 184 mg/L; see Table 44). The ORNL/MWL extractions contained the highest average K concentrations, 521 mg/L (significantly greater at $P < 0.05$ than all other media). Average K concentrations extracted by the other media (acetate, carbonic acid, and de-ionized water) were not significantly different from each other. Unlike Ca, there were significant ($P < 0.05$) decreasing K concentrations in extractions conducted at 2.5, 5, 10, and 20:1 liquid/solid ratios, respectively (Table 44).

As one would expect, Na extractions from the API/incinerator ash nearly mimicked those of K extractions (Fig. 79 as compared to Fig. 78). In both cases, the rotary-batch extractions contained higher concentrations ($P < 0.05$) than upflow-column extracts (Table 44). However, with respect to Na, the acetate buffer extractions removed greater quantities ($P < 0.05$) than the ORNL/MWL extractions, which was not the case with K. De-ionized distilled water and carbonic acid extracts contained similar Na concentrations.

One would also expect the extraction of Sr to be similar to that of Ca. However, there was little similarity in their extraction by the two extraction procedures or in their liquid/solid ratios. For example, significantly ($P < 0.05$) higher concentrations of Ca were observed in upflow-column extracts than in rotary-batch extracts, but there were no significant differences in Sr concentrations extracted by the two procedures (Table 44). There appears to be an interaction between extraction procedure and liquid/solid ratio using carbonic acid as the extracting medium (Fig. 80); however, the absolute difference in Sr concentrations, at any specific liquid/solid ratio, between the two extraction procedures is relatively small. Perhaps the greatest difference between Ca and Sr extractability is the decreasing concentrations ($P < 0.05$) of Sr extracted on increasing liquid/solid ratios, which did not occur with Ca extractions (Table 44).

The extraction of naphthalene from API/incinerator ash appears to be much less dependent on liquid/solid ratios (Fig. 81) than were the extractions of inorganics. For some reason, there were significantly lower ($P < 0.05$) concentrations in extracts at the liquid/solid ratio of 5:1 than at the other liquid/solid ratios (Table 44). The rotary-batch procedure extracted higher concentrations ($P < 0.05$) than the upflow-column, and the carbonic acid medium extracted higher concentrations of naphthalene than any of the other media. Of the media tested, the ORNL/MWL medium extracted the least. However, none of the extractions (combination of procedure and medium) contained naphthalene in concentrations as high as the lower 95% confidence level for MLC (0.45 mg/L; Table 37) determined from the field leachates. The lower levels of naphthalene found in the laboratory extracts suggest that some naphthalene is either not completely extracted from the waste or a portion is lost during the extracting procedure. The nonpolar nature of this compound may have caused losses to occur by adsorption, either on the walls of the extracting vessel or during the collection of the filtrate. Volatility of naphthalene may also have contributed to losses. Note that the column procedure contained significantly lower ($P < 0.05$) concentrations of naphthalene in the extracts as compared to the rotary-batch extracts. This is interesting in that extracts from the column procedure were collected directly into Tedlar bags (closed system), while the rotary-batch extracts were not, possibly allowing the naphthalene to be lost by evaporation, implying that extraction by the rotary-batch procedure is more effective than the closed upflow-column procedure.

Dichloroethylene Still Bottoms

The extraction of dichloroethane, trichloroethane, and trichloroethylene from the dichloroethylene still bottoms (Figs. 82 to 84) was independent of the type of procedure (upflow-column or rotary-batch) or liquid/solid ratio used (Table 44). The ORNL/MWL extracts, contained significantly higher ($P < 0.05$) concentrations of trichloroethane and trichloroethylene than the other extracting media. Highest concentrations of dichloroethane were observed in carbonic acid extracts (not significantly different from the ORNL/MWL extracts but significantly higher [$P < 0.05$] than those in acetate or water [Table 44]). All extracts contained concentrations less than their respective MLC 95% confidence levels (Table 37), except the extractions for trichloroethylene using ORNL/MWL (both column and batch procedures) and distilled water at the 5:1 liquid/solid ratio; both of these were within the 95% confidence levels for trichloroethylene (67 to 162 mg/L). The high volatility of these organic compounds, coupled with the fact that all are soluble at the levels measured, indicates that considerable care must be exercised to prevent evaporative losses during extraction. This is quite difficult experimentally, both in terms of handling the waste (e.g., storage--although these wastes were stored in closed containers that were kept under refrigeration, losses by evaporation no doubt occurred in weighing samples and transferring them to the extraction vessels) and during extract generation. Evaporative losses directly from the filtered extracts appeared not to be a factor because there were no significant differences noted between the extract concentrations of these volatile organic compounds in upflow-column extracts (those collected directly into the Tedlar bags) and those in rotary-batch extracts (Table 44).

The rotary-batch procedure extracted much higher concentrations of HCB than the upflow-column procedure did at liquid/solid ratios 2.5 and 5:1. Higher concentrations were observed at low liquid/solid ratios (2.5 and 5.0 as compared to 10 and 20:1 ratios) only with rotary-batch extractions (i.e., few differences in extract concentration were measured in column extractions across liquid/solid ratios; see Fig. 85). Analyses for significant differences among main effects (procedures, media, and liquid/solid ratios) showed significant differences ($P < 0.05$) in extract concentrations at the 2.5:1 ratio as compared to the other liquid/solid ratios; however, the higher concentrations at 5:1 were not significantly different from 10 and 20:1 ratios (Table 44). Acetate extracts contained the highest HCB concentrations (significant at $P < 0.05$). Average concentrations among the remaining media (carbonic acid, water, and ORNL/MWL) were not statistically different.

Paint Sludge

The extract from the sodium acetate pH 5 buffer at a liquid/solid ratio of 2.5:1 contained the highest Ba concentration (4 mg/L; Fig. 86) of all extractions used. When all media and liquid/solid ratios are considered, the rotary-batch procedure extracted significantly ($P < 0.05$) higher concentrations of Ba than the upflow-column procedure (average concentrations 1.8 and 0.98 mg/L, respectively; see Table 44). However, when using only the ORNL/MWL as the extracting medium, the upflow-column procedure extracted higher concentrations of Ba than did the rotary-batch procedure (Fig. 86). Because of the relatively close upper and lower 95% confidence levels (2.2 to 2.9 mg/L; Table 39) for the MLC concentration of Ba, only three extractions (the rotary-batch at liquid/solid ratio of 2.5 using carbonic acid or de-ionized distilled water and the upflow-column at a liquid/solid ratio of 5 with ORNL/MWL) produced extracts having concentrations within the Ba MLC 95% confidence levels. The acetate buffer and ORNL/MWL extracts contained higher concentrations ($P < 0.05$) of Ba than did the de-ionized distilled water and carbonic acid extracts (Table 44).

Zinc is present at much higher concentrations in paint waste leachates than is Ba (220 as compared to 2.5 mg/L, MLC target concentrations). As with Ba, the concentrations of Zn extracted with the rotary-batch procedure were statistically significant ($P < 0.05$), higher (189 mg/L) than those from the upflow-column (132 mg/L) procedure (across all media and liquid/solid ratios; see Fig. 87 and Table 44). At a liquid/solid ratio of 2.5, the rotary-batch procedure extracted higher concentrations of Zn than the MLC upper 95% confidence levels for Zn (274 mg/L; Table 37); the single exception was when the ORNL/MWL was used as an extraction medium. The MLC concentrations for Zn (between the upper and lower 95% confidence levels) could be simulated using the rotary-batch procedure and three of the four media (not acetate) at a liquid/solid ratio of 5. At a 2.5 liquid/solid ratio, the rotary-batch procedure with ORNL/MWL would also produce extracts with Zn concentrations within the MLC 95% confidence levels. These same concentrations of Zn could be attained using the upflow-column procedure with three of the four media (not ORNL/MWL) at a 2.5 liquid/solid ratio. With ORNL/MWL as a medium, a 5:1 liquid/solid ratio would have to be used to yield Zn extract concentrations similar to the MLC 95% confidence levels (Fig. 87). As with Ba, de-ionized distilled water extracts contained the lowest Zn concentrations among the four media, and concentrations increased significantly ($P < 0.05$) with decreasing liquid/solid ratios (Table 44).

Ethoxyethanol was present in paint sludge extracts at concentrations (average concentrations of about 2000 mg/L) higher than any inorganic element or organic compound (Table 44). The solubility of this compound should be unaffected by the changes in media and should depend primarily on liquid/solid ratio. However, the concentration varied depending on the extracting procedure, media, or liquid/solid ratio used (Fig. 88). Statistically, however, there was not a significant difference in ethoxyethanol levels using upflow-column or rotary-batch procedures across all media and liquid/solid ratios (Table 44). The differences in concentrations at 2.5 and 5:1 liquid/solid ratios using the acetate pH 5 buffer and carbonic acid (Fig. 88) are obviously major differences (as great as 400 mg/L) and if tested for significance would probably be significant at the 5% level (note that testing for statistically significant differences between the 32 treatment means and potential interactions among procedure, media, and liquid/solid ratios was not undertaken at this time). The ORNL/MWL medium extracted concentrations of ethoxyethanol that were twice those extracted by the other media (significant at the $P < 0.05$ level; see Table 44). A similar, but even more pronounced, effect of preferential extraction by ORNL/MWL was observed in the extraction of ethoxyethyl acetate from the paint sludge (discussed below). At the 2.5 liquid/solid ratio, extracts from both the column and the batch procedures using ORNL/MWL, in fact, exceeded the MLC upper 95% level (Fig. 88 and Table 37) for ethoxyethanol. However, at the 5:1 liquid/solid ratio, extract concentrations were within the MLC levels using any of the media (see Fig. 88 for the specific procedure and/or liquid/solid ratio). This probably indicates the ability of MWL to disperse the waste, because the solubility of ethoxyethanol is essentially infinite.

The differences in concentrations of ethoxyethyl acetate between upflow-column and rotary-batch extracts (using the acetate pH 5 buffer as a medium; Fig. 89) appear to be great; however, similar differences were not measured using the other media, and statistical analyses across all media and liquid/solid ratios indicated no significant differences (Table 44). Significantly higher concentrations were observed in extracts made at 2.5 on 5:1 liquid/solid ratios as compared to 10 or 20:1 ratios. As mentioned above, the ORNL/MWL extracts contained much higher concentrations (at least twice those of the next highest medium--water) than all of the other extracting media tested (significant at $P < 0.05$). The MLC concentrations (within the 95% confidence levels, 1307 and 2476 mg/L) could only be attained using ORNL/MWL at 2.5 and 5:1 liquid/solid ratios with the rotary-batch extractor or with the upflow-column at the 2.5:1 ratio (Fig. 89).

Extraction of the paint sludge for toluene resulted in extract concentrations that were uniformly low (<10 mg/L) showing little preference for type of procedure, media, or liquid/solid ratio (Fig. 90). However, statistical analyses detected differences between media ($P < 0.05$), revealing that the de-ionized distilled water and carbonic acid extracted higher concentrations than did acetate and/or ORNL/MWL (Table 44). This may result from enhanced solubility of toluene in low ionic strength media. None of the laboratory extracts contained concentrations of toluene that were within the MLC 95% confidence levels (23 to 56 mg/L; Table 37). Toluene, like the halocarbons mentioned earlier, has appreciable volatility, particularly at low concentrations. Thus, the levels measured in the extracts might be low because of evaporative losses.

Figure 91 shows major differences in extract concentrations of xylenes using upflow-column and rotary-batch procedures with acetate (at liquid/solid ratios of 2.5 and 5:1), carbonic acid (all liquid/solid ratios), and de-ionized distilled water (liquid/solid ratios less than 20:1) as the extraction media. However, these differences were not great enough to show significant differences ($P < 0.05$) across all media and liquid/solid ratios (Table 44). The extraction of the xylenes and toluene from the paint sludge may have been solubility limited because no significant differences were observed in extract concentrations on increasing liquid/solid ratios (Table 44). The ORNL/MWL extracted higher concentrations of xylenes than any other medium--not significantly different than acetate pH

5 buffer, though significantly different ($P < 0.05$) than the other two media. Only for the rotary-batch procedure with the acetate pH 5 buffer, at 2.5 liquid/solid ratio, was the average extract concentration of xylenes within the MLC 95% confidence range (286 to 942 mg/L; Table 37). Again, evaporative losses may have played a role in keeping extract concentrations low.

Electroplating Waste

Concentrations of Ba in leachates from the electroplating waste (0.34 to 0.60 mg/L as the MLC lower and upper 95% confidence levels; Table 37) were much lower than Ba concentrations in leachates from the paint sludge (MLC levels, 2.0 to 2.6 mg/L; Table 37). One of the reasons for this lower concentration, other than its lower total concentration in the electroplating waste, as compared to the paint sludge, is the low water solubility of Ba at pH values greater than 7.5 to 8.0. Note that the peak Ba concentration in the electroplating waste did not occur until the pH of the leachate from the lysimeter test facility had reached 7.0 ± 0.2 (liquid/solid ratio of 6.3; see Fig. 74). The point here is that the high alkalinity (pH values near 9 for de-ionized distilled water extracts) limits solubility of the Ba in the electroplating waste extracts. For example, the only laboratory extracts that contained appreciable concentrations of Ba were those that had pH values similar to the leachates from the field lysimeter at MLC target concentrations. The same pH effect is seen on the removal of Ni and Zn from the electroplating waste (to be discussed later). It is interesting that only the ORNL/MWL extracting medium, using the upflow-columns at liquid/solid ratios of 10 and 20:1, contained Ba concentrations between the MLC lower and upper 95% confidence levels (Fig. 92). In contrast to the B extracted from the electroplating waste (as well as any of the other MLC inorganic elements extracted from the other wastes), higher Ba concentrations were observed in larger liquid/solid ratios of 10 and 20 than the 2.5 and 5:1 ratios (Table 44). This, again, is a result of the final pH of the extract (Table 45). At high liquid/solid ratios, sufficient volumes of acetate, carbonic acid, and/or ORNL/MWL have been added to lower the pH of the suspension so that the Ba is solubilized and extracted into the solution.

The rotary-batch procedure extracted higher concentrations of B from the electroplating waste than the upflow-column method with three extracting media (not ORNL/MWL; Fig. 93). However, no extraction method (procedure and/or medium) produced extracts in which the B concentrations exceeded MLC's lower 95% confidence level (139 mg/L). Boron in the electroplating waste must be in the form of a carbonate or other alkaline mineral that weathers on interaction with an acidic medium. Note the significantly higher ($P < 0.05$) concentrations of B extracted by the acidic media over those extracted by the de-ionized distilled water (Table 44).

The ORNL/MWL extracted higher concentrations of Mn than any of the other extracting media (Table 44). Concentrations of MN in the de-ionized distilled water extracts were below detection by ICP (Fig. 94). Using ORNL/MWL, upflow-column extractions contained higher Mn concentrations than rotary-batch extractions. This relationship prevailed, however, only at the lower liquid/solid ratios for the 0.1 M sodium acetate and carbonic acid extractions. Over all media and liquid/solid ratios, there were no significant differences between Mn concentrations obtained by upflow-column or rotary-batch extraction procedures (Table 44). Higher Mn concentrations ($P < 0.05$) were observed in extracts made at larger (10 and 20:1 as compared to the lower 2.5 and 5:1) liquid/solid ratios.

The acidic extracting media (acetate pH 5 buffer and the ORNL/MWL) generated extracts containing factors of 10 and 100 times higher Ni concentrations than carbonic acid and de-ionized distilled water (see Fig. 95). Average Ni concentrations, across both extraction procedures and all liquid/solid ratios, were 85.5, 71.2, 27.3, and 0.45 mg/L respectively for acetate, ORNL/MWL, carbonic acid, and water. The higher concentrations of Ni extractions at liquid/solid ratios of 10 and 20:1 as compared to 2.5 and 5:1 result from the lower pH at these higher liquid/solid ratios when using the acidic extracting media (acetate, ORNL/MWL, and to some extent carbonic acid; see Table 45).

The extraction of K from the electroplating waste (Fig. 96) was unlike the K extraction from the API/incinerator ash. With the electroplating waste, there were no significant differences in K extract concentrations between the upflow-column or the rotary-batch extraction procedure. In the API/incinerator ash extracts, K was higher ($P < 0.05$) in the rotary-batch extracts than in the upflow-column extracts (Table 44). The ORNL/MWL extracts, from both wastes, contained the highest K concentrations ($P < 0.05$). As expected, K concentrations decreased on increasing liquid/solid ratios; however, the difference between 10 and 20:1 ratios was not statistically significant. (On the other hand, K extractions from API/incinerator ash were significantly different, $P < 0.05$, at all liquid/solid ratios.)

The extraction of Na from the electroplating waste followed a pattern almost identical to the extraction of Na from the API/incinerator ash. For example, the same pattern held for differences between upflow-column and rotary-batch, the effect of the extracting media, and liquid/solid ratios (Table 44). For the electroplating waste, the differences in Na concentrations between upflow-column and rotary-batch procedures were greatest at low liquid/solid ratios (2.5 and 5:1) using acetate, carbonic acid, and de-ionized distilled water as the extracting media (Fig. 97). The average Na concentration in all laboratory extracts (1561 mg/L) from the electroplating waste was about 30% larger than the average Na concentration of all laboratory extracts from the API/incinerator ash (1189 mg/L). The total Na content of the electroplating waste, however, was only approximately 3% greater than the API/incinerator ash.

Strontium extractability from the electroplating waste was unlike the Sr from the API/incinerator ash. For example, Sr concentrations in the electroplating waste extracts were 20 times lower than Sr concentrations in the API/incinerator ash extracts (0.30 and 6.0 mg/L, respectively), reflecting the lower total Sr content of the two wastes (<19 and 260 mg/kg, respectively). De-ionized distilled water extracts of the electroplating wastes were below ICP detection limits (Fig. 98). Perhaps the greatest difference in the pattern of Sr extraction from the electroplating wastes as compared to Sr from the API/incinerator ash was the higher ($P < 0.05$) Sr concentrations in electroplating waste extracts on increasing liquid/solid ratios, suggesting increased solubilization on acidification of the waste. Strontium concentrations in the API/incinerator ash extracts decreased on increasing liquid/solid ratios, suggesting that Sr was present in the waste as a water-soluble compound. On the other hand, the pattern of Sr release from the electroplating wastes suggested that the Sr was present as a carbonate mineral!

With respect to the extraction of Zn from the electroplating waste, the sodium acetate buffer produced extracts containing the highest overall Zn concentrations (average 175, as compared to 79.1, 36.6, and 0.41 mg/L, respectively, for ORNL/MWL, carbonic acid, and water as extracting media). Upflow-column extracts contained higher Zn concentrations than did rotary-batch extracts ($P < 0.05$), particularly at low liquid/solid ratios (see Fig. 99). Sodium acetate buffer in an upflow-column produced extract concentrations of Zn in excess of the MLC 95% confidence levels (107 to 191 mg/L) at all liquid/solid ratios. As mentioned earlier for Ba and Ni, the pH of the extracting medium (Table 45) is the most important parameter governing the extraction of Zn from the electroplating waste.

One of the principal objectives in conducting the laboratory extraction experiments was to compare the effectiveness of extraction by rotary-batch and upflow-column procedures. There were significant ($P < 0.05$) differences in extract concentrations between these two extraction procedures for 13 of the 25 target constituents (average concentrations of rotary-batch and upflow-column extracts across all extraction media [acetate, carbonic acid, de-ionized distilled water, and ORNL/MWL] and liquid/solid ratios [2.5, 5, 10, and 20:1]). Of the 13 target constituents where statistically significant ($P < 0.05$) differences between procedures were detected, the rotary-batch extracted higher concentrations for 10 of the 13 target constituents, suggesting that the rotary-batch extraction procedure is more aggressive in extraction of target constituents than the upflow-column extraction procedure.

Bi-Sequential Extraction

For an extraction method to simulate MLC target values, the liquid/solid ratio should be as low as possible, and at the same time, it should have a final pH similar to the pH of the ORNL/MWL. Of the 25 MLC lysimeter target concentrations, 18 were observed at a liquid/solid ratio less than 5:1 (Table 37). The average liquid/solid ratio for all 25 target concentrations was 3.42 ± 3.19. The paint sludge had the lowest average liquid/solid ratio (1.26 ± 0.61) for its six target concentrations (four organic compounds and two inorganic elements). The principal reasons for such a low average target liquid/solid ratio were that the four organic compounds readily leached from the waste and that the pH of the initial waste leachate was below that of the control ORNL/MWL (range between 5.5 and 6.0; see Fig. 74). Thus, leaching of the target constituents in the paint sludge was not pH dependent. On the other hand, the average liquid/solid ratio of the target inorganic elements in the electroplating waste was high (5.16 ± 4.39) and ranged greatly, from 0.69 for B and Na to 14 for Zn. The leaching of Ni and Zn in the electroplating waste was highly dependent on lowering the leachate pH to the range of 7.5 to 6.0. To extract high concentrations of readily soluble constituents (e.g., Zn and the organic compounds in the paint sludge) and the elements that are highly dependent on a low extracting pH (e.g., Ni and Zn in the electroplating waste), a bi-sequential laboratory extraction method was developed. The method consisted of a rotary-batch procedure at an initial liquid/solid ratio of 5:1, followed by adding a fresh 100 g of waste to the filtered extract and extracting for another 18 h to make a final liquid/solid ratio of 2.5:1. The medium chosen was a sodium acetate pH 5 buffer. For the electroplating waste, a preliminary experiment revealed that it was necessary to use a strength of 0.25 M acetate in such a bi-sequential procedure to bring the final extract to a pH of less than 7.5. For the other three wastes, a 0.1 M sodium acetate buffer was used.

Comparisons of Laboratory Extractions to Lysimeter Target Concentrations

The purpose of the research was to develop a laboratory extraction method (combination of procedure, media, and liquid/solid ratio) that simulated target concentrations of potential inorganic and organic contaminants present in solid waste leachates as a result of the co-disposal of municipal and industrial wastes (95:5 ratio) in landfills. As discussed earlier, the target constituents were those 16 inorganic elements and 9 organic compounds that were measured in the industrial waste leachates in greater quantities than in the control MWL leachates. Five sets of target concentrations--MLC, AMC8, AMC20, AMC40, and IAC8--were established using three alternative approaches. These approaches included (1) the MLC observed during the 97-d leaching, (2) an average maximum concentration (AMC) for a specific leaching period, expressed as a liquid-to-solid ratio bracketed around the MLC, and (3) an integrated average concentration (IAC) expressed over the initial 8:1 liquid-to-solid ratio. These target values and their corresponding upper and lower 95% confidence levels are displayed in Tables 37 to 41.

The laboratory study involved 32 different extractions, each replicated twice (two procedures--upflow-column and rotary-batch; four extracting media--0.1 M acetate pH 5 buffer, carbonic acid, de-ionized distilled water, and leachate from the field lysimeter, ORNL/MWL; and four liquid/solid ratios--2.5, 5, 10, and 20:1). The data from the laboratory study were combined with the data from the lysimeter study for each targeted chemical/waste combination.

The difference between the laboratory concentrations and their respective target concentrations was defined as follows:

Difference = [abs(TC - LC)/TC] × 100,

where

TC is the target concentration,
LC is the laboratory concentration, and
abs( ) is the absolute value and the difference is expressed in percent.

An average difference between the target concentration and the laboratory concentration was then determined for each of the 32 treatments and each of the 25 target constituents. An overall average difference was then determined for each of the categories: (1) inorganic, (2) organic, and (3) inorganic and organic target constituents for each of the five sets of target concentrations (MLC, AMC8, AMC20, AMC40, and IAC8). These average differences were then ranked from the lowest to the highest (Tables 46 to 60). For the 32 replicated extraction methods (all but the EP and bi-sequential extraction methods were duplicated), significant differences between extractions could be determined in an analysis of variance testing procedure (Statistical Analysis System [SAS] software package [SAS Institute 1982]).

MLC Target Concentrations: The laboratory extraction procedure that came closest to simulating MLC inorganic target concentrations was an upflow-column using ORNL/MWL at a liquid/solid ratio of 10:1. It, however, still generated laboratory extracts that contained concentrations whose average difference relative to the target concentrations was slightly greater than 50%. Its closest laboratory concentration was within 10.8% of the target value, and its most distant concentration was 97.7% of the target value. The EP came within 1.9% of the target value at least once (see Table 46 for minimum difference, but its overall average difference was almost 20% greater than the best ranked method (average difference of 69.9% for the EP as compared to 50.2% for the first ranked method).

The ORNL/MWL was also the prevailing medium that best simulated MLC organic target concentrations (Table 47). For example, of the top eight ranked extraction methods (those not significantly different at $P < 0.05$), ORNL/MWL was used in five of the extraction methods. Within these eight methods, five were rotary-batch procedures and three were upflow-column extractions. The prevailing liquid/solid ratio was 2.5:1. Excluding ORNL/MWL as an extracting media in these top eight methods leaves either acetate, carbonic acid, or de-ionized distilled water in a rotary-batch extraction at a liquid/solid ratio of 2.5:1, suggesting that use of any of these extracting media in a rotary-batch extractor at a liquid/solid ratio of 2.5:1 would effectively model MLC organic target concentrations. The EP ranked very poorly (32 of 34 methods) in simulating MLC organic target concentrations (Table 47).

The top six ranked methods that simulated MLC inorganic and organic target concentrations used ORNL/MWL as an extracting medium. Of the top 10 ranked methods, sodium acetate and carbonic acid extraction media in rotary-batch extraction procedures at liquid/solid ratios of 2.5 to 10:1 were the only synthetic media (Table 48). All of these extraction procedures, using acetate or carbonic acid as an extraction medium, were statistically equivalent in ranking to the fourth procedure; however, they were significantly lower ($P < 0.05$) than the top three methods. The poorest ranked method was the bi-sequential extraction, followed by extractions utilizing de-ionized distilled water as the extracting media at liquid/solid ratios of 10 and 20:1.

AMC8 Target Concentrations: Carbonic acid was the extraction medium used in all of the top four ranked extraction methods to simulate AMC8 inorganic target concentrations (Table 49). Three of the four methods used the upflow-column procedure at liquid/solid ratios of 5, 10, and 20:1. These four top ranked extraction methods, however, are not significantly different than the first 13 ranked extraction methods. Again, the bi-sequential method was ranked last. The EP was ranked 20, with an average percent difference of the target concentrations of almost 100% as compared to 44.4% for the first ranked extraction method.

Relative to simulating AMC8 organic target concentrations, no specific extracting medium or procedure prevailed in the top five ranked methods (Table 50). Noticeably absent, however, were ORNL/MWL and de-ionized distilled water. The high variability in extraction replicates for organic compounds precludes demonstrating statistically significant differences among treatments. For example, the best ranked method (lowest average difference, 57.4%) was not significantly different than the method ranked 32 (average difference, 259.9%).

Perusal of Table 51 immediately projects the utility of carbonic acid as an extracting medium to simulate AMC8 inorganic and organic target concentrations. The top five ranked methods, however, are not statistically different than the first 26 ranked methods, largely because of the high variability in the extraction of organic compounds from the wastes. It is quite evident, however, that to simulate AMC8 inorganic and organic target concentrations carbonic acid would be chosen over the other extraction media tested, regardless of the statistical interpretations. Because of the relatively small differences in ranking of the first five extraction methods (range in average differences from 54.8 to 64.4%), the choice of extracting procedure would be dictated by differences in operational conditions of the two procedures (upflow-column and rotary-batch). As stated earlier in the Methods and Materials section, numerous operational problems are associated with the upflow-column (e.g., some wastes dictate very slow flow rates because of their low hydrologic conductivities). Upflow-columns also require expensive pumps and generally require more training of laboratory personnel to operate than do rotary-batch extractors, which precludes their selection when rankings between the two procedures are as close as those presented in Table 51. Choice of the specific liquid/solid ratio appears not to be of major importance in that the first five ranked methods (those using carbonic acid) employed 5, 10, and 20:1 liquid/solid ratios.

AMC20 Target Concentrations: AMC20 target concentrations were determined in a manner similar to AMC8 target concentrations. The only difference was that the liquid/solid ratio bracketing the MLC was 20 instead of 8. Consequently, for most leaching curves, AMC20 target concentrations will be lower than AMC8 target concentrations. The two will be equal when the leaching rate (concentration over time) is constant over the bracketed liquid/solid ratios (8 and 20:1). In special cases, the AMC20 target concentration can be more than that of the AMC8. For example, if the MLC is observed at a liquid/solid ratio lower than 4, the AMC8 is, by default, the integrated average concentration over the first 8:1 liquid/solid ratio. In the leaching of K and Sr from the electroplating waste, concentrations of K and Sr were observed in control leachates at values higher than in the industrial waste leachate (Figs. 40 and 44). A number of possibilities exist that could cause the control leachates (in this case, those MWLs passing through the sand) to be higher than the industrial waste leachates, especially early in the leaching curve.

The principal hypothesis for the higher concentrations in the control leachates is that the sand used in the control columns was a very poor attenuator of K and Sr in the MWL. On the other hand, in the columns with the electroplating waste, which contained very high concentrations of leachable Na (see Fig. 42), the Na was leached preferentially to K and Sr, thus attenuating the mobility of K and Sr originating in the MWL. An even more pronounced demonstration of a similar effect is the leaching of Mn from the electroplating waste (Fig. 41). The Mn concentrations in control leachates (at liquid/solid ratios of 3 to 4) were higher than in the industrial waste leachates until the pH of the leachate was low enough to allow the movement of Mn through the waste (Fig. 74).

For K and Sr leaching from the electroplating waste, the average liquid/solid ratio for the MLC was less than or very close to 4 (Table 37). Also, the integrated average concentration over the first 8:1 liquid/solid ratio was determined for each leaching curve, not for an average of the four replicates. Thus, for K and Sr in the electroplating waste, the AMC20 values reflect higher concentrations than the AMC8 values because the difference between the concentrations of the target constituents in the leaching of the industrial wastes and the controls became greater at large liquid/solid ratios (between liquid/solid ratios of 4 and 10; see Figs. 40 and 44). In general, AMC20 target values will be lower than AMC8 target values (however, there will be exceptions). Thus intuitively, less aggressive extraction methods (those that use weaker extracting media and/or higher liquid/solid ratios) will tend to produce concentrations closer to AMC20 target values than more aggressive extraction methods.

Of the first five ranked extraction methods to simulate AMC8 inorganic target concentrations (Table 49), four were ranked in the first five ranked methods to simulate AMC20 inorganic target concentrations (Table 52). De-ionized distilled water in an upflow-column at a liquid/solid ratio of 20:1 replaced carbonic acid in an upflow-column at a liquid/solid ratio of 5:1. Four of the five methods utilized the upflow-column procedure, and four of the five methods (irrespective of procedure) were conducted at liquid/solid ratios of 20:1. Statistically, there were no differences ($P < 0.05$) in the ranking of the top 10 methods (Table 52). Over all extractions there was a prevailing tendency of lower liquid/solid ratios on decreasing rank (higher average difference).

Only one of the top five ranked extraction methods for simulating AMC8 organic target concentrations (sodium acetate in rotary-batch at a liquid/solid ratio of 20:1) was in the top five ranked methods to simulate AMC20 organic target concentrations (Table 53). Unlike the preferred procedure for AMC20 inorganic target concentrations (the upflow-column), four of the five highest ranking methods for AMC20 organic concentrations used rotary-batch procedures. Again, because of the high variability between laboratory extraction replicates, there was a wide range in distinguishing significant differences ($P < 0.05$) among extractions (Table 53). Compared to previously discussed rankings, the EP ranked relatively well (8 out of the 34).

The dominance of carbonic acid as an extraction medium for AMC20 inorganic and organic target concentrations (Table 54) did not occur as it had for AMC8 inorganic and organic target concentrations (Table 51). In the AMC20 top five rankings, the carbonic acid extractions (a liquid/solid ratio of 10:1 in the rotary-batch procedure and a liquid/solid ratio of 5:1 in the upflow-column procedure) that prevailed in the AMC8 top five rankings were replaced with de-ionized distilled water in upflow-column procedure at a 20:1 liquid/solid ratio and sodium acetate pH 5 buffer at a 20:1 liquid/solid ratio. Both of these latter extractions, because of their high liquid/solid ratios, tend to be less aggressive extractions than those at the lower liquid/solid ratios. The upflow-column extraction procedure was utilized more times (four out of the five) than the rotary-batch procedure. Statistically, there were no significant differences in the rankings of the top 24 extraction methods.

AMC40 Target Concentrations: The relative rankings of the extraction methods to simulate AMC40 target concentrations will be discussed in a comparative sense; however, keep in mind that the extrapolation to liquid/solid ratios in excess of the experimental data (all less than 20:1 liquid/solid ratios) is highly speculative.

The trend in type of extracting medium and liquid/solid ratio that best simulated AMC40 target concentrations was very similar to that observed for the AMC20 target concentrations [i.e., the best extraction media were generally carbonic acid and distilled water (weak extracting media relative to MWL or acetate) at large liquid/solid ratios (20:1)]. Perhaps the most significant difference between the two target sets (AMC40 and AMC20) was that the best ranked method (carbonic acid at liquid/solid ratio of 20:1) for AMC40 inorganic targets and AMC40 inorganic and organic targets (Tables 55 and 57) averaged approximately 100% from the targets, while the best ranking extraction methods for AMC20 target concentrations were approximately 50% from the target concentrations (Tables 52 to 54). On the other hand, the average difference (56%, Table 56) for the best extracting method for AMC40 organic target concentrations was not appreciably different than the best ranking extraction method (sodium acetate, rotary-batch at 20:1 liquid/solid ratio) for AMC20 organic target concentrations (Table 57). The principal reason for the ineffectiveness in simulating the inorganic target concentrations is probably caused by the much lower AMC40 inorganic target concentrations (chiefly Na in both the API/incinerator ash and the electroplating waste) relative to the AMC20 inorganic target concentrations (see Table 61). The differences between AMC20 and AMC40 organic target concentrations (Table 61) are not appreciable (especially for the organic compounds in the dichloroethylene still bottoms).

IAC8 Target Concentrations: The major differences in IAC8 and AMC8 target values are the lower IAC8 concentrations for Ni and Zn in the electroplating waste (respectively, 80 and 49 mg/L as compared to 147 and 149 mg/L for AMC8). The IAC8 value reflects only the initial part of the leaching curve, whereas the AMC8 value represents that part of the curve when the acid pH conditions of the MWL mobilized the Ni and Zn from the waste. Generally, the values for the IAC8 target concentrations are either the same or lower than for AMC8 concentrations, but in three cases IAC8 target concentrations were slightly higher than the AMC8 values. They are Ca and Mo in the API/incinerator ash and dichloroethane in the dichloroethylene still bottoms. These small differences (5 mg/L for Ca, 0.06 mg/L for Mo, and 4 mg/L for dichloroethane) resulted as a consequence of the form of the leaching curve and the fit to that curve. The major contributing factor was that for most of these target constituents the 4:1 liquid/solid interval on the leeward side of the (on right hand side) AMC8 leaching curve contained less material than the analogous side of the IAC8 leaching curve. Close inspection of these leaching curves will indicate this.

The most notable observation relative to the ranking of the extraction methods to simulate IAC8 target concentrations is that de-ionized distilled water extracts dominated the first 10 ranking positions (Tables 58 to 60). This was particularily true for the inorganic rankings. For example, seven of the eight extraction methods that used de-ionized distilled water as an extracting medium were in the top 10 ranked methods to simulate IAC8 inorganic target concentrations. In retrospect, this is not surprising: (1) the pH of the leachates from the API/incinerator ash and the electroplating waste is still relatively high during this leaching interval (see Fig. 69) and (2) the time over which this leaching interval was conducted is relatively short (approximately 33 d). Under these conditions, the interactions of the MWL on the wastes have been predominantly the same as the interaction of distilled water (i.e., it leaches out the water-soluble constituents). Continued interaction of MWL produces MLC target concentrations whose liquid/solid ratios reflect increased solubilization (liquid/solid ratios greater than 8) of the target constituents. For example, with respect to the leaching of Ba, Sr, Ni, and Zn (from the electroplating waste), dichloroethane and trichloroethylene (from the dichloroethylene still bottoms), and Ca, Mo, and to a certain extent Sr (from the API/incinerator ash), the liquid/solid ratio of 8:1 represents a relatively small fraction of the total moss leached. Possibly to be of more utility, the IAC approach should be extended to a larger liquid/solid ratio (e.g., 20:1, which for most target constituents would be the same as AMC20).

SUMMARY AND CONCLUSIONS

A target constituent was defined as any inorganic element or organic compound that exhibited a distinct concentration maximum over the 79-d leaching period and whose total mass leached from the industrial waste was greater than that leached in the control municipal waste leachate (MWL). A total of 25 target constituents (16 inorganic elements and 9 organic compounds) were identified in the leachates of the following industrial wastes:

API/Incinerator Ash

The inorganic elements Ca, Cr, K, Mo, Na, and Sr, and the organic compound naphthalene.

Dichloroethylene Still Bottoms

No inorganic chemicals, but four organic compounds: dichloroethane, trichloroethane, trichloroethylene, and hexachlorobutadiene.

Paint Sludge

The inorganic elements B and Zn, and the organic compounds ethoxyethanol, ethoxyethyl acetate, toluene, and xylenes.

Electroplating Waste

The inorganic elements B, Ba, K, Mn, Na, Ni, Sr, and Zn, but no organic compounds.

Five sets of target concentrations were established using three basic criteria. The first criterion involved the identification of a maximum leachate concentration (MLC) over the leaching period. The second criterion identified an average maximum concentration (AMC) over a specific leaching interval bracketed around the MLC. The third and final criterion was an integrated average concentration (IAC8) taken over the first eight liquid/solid ratios.

The intent of the research was to establish target concentrations using all three criteria and then to rank the various laboratory extraction methods as to which method most closely simulated the particular target concentration. Five sets of target concentrations

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for the 25 inorganic and organic constituents were established. The five sets were as follows:

1. MLC--measured in lysimeter leachates over 79 d of leaching,
2. AMC8--measured in an 8:1 liquid/solid leaching interval that bracketed the MLC measured in lysimeter leachate,
3. AMC20--measured in a 20:1 liquid/solid leaching interval that bracketed the MLC measured in lysimeter leachate,
4. AMC40--measured in a 40:1 liquid/solid leaching interval that bracketed the MLC measured in lysimeter leachate, and
5. IAC8--leached over the first 8:1 liquid/solid ratio.

The rationale used to select the liquid/solid leaching intervals (those used in AMC and IAC) was based on the Kimmell and Friedman draft manuscript. The 8:1 liquid/solid ratio was selected because a perusal of preliminary data relating to the quantity of leachate that moved through a municipal waste landfill indicated that an 8:1 liquid/solid ratio represented a period of leaching equivalent to 1 to 3 years, depending on the assumptions regarding meteorologic and geologic conditions and landfill design. The 20:1 liquid/solid ratio was used because it represents a significantly longer leaching period, and is consistent with the liquid/solid ratio currently being used in the Resource Conservation and Recovery Act (RCRA) extraction procedure (EP). The 40:1 ratio was included primarily to illustrate possible long-term leaching characteristics of the various target constituents.

Target values for MLC were defined as the average maximum concentration measured in lysimeter leachates over the 79 d in the four replicated industrial waste leachates. Control concentrations for each replicate were subtracted at the MLC liquid/solid ratio. Target concentrations for AMC8, AMC20, AMC40, and IAC8 were determined as follows. First, the accumulative leaching curves for each of the 25 target constituents were fitted, using the best fit curve from a selection of four basic single-variable models. Fitting of the accumulative leaching curves to one of these four models provided equations so that quantities of the leached constituents could be calculated over discrete liquid/solid ratios. AMC target concentrations were calculated for liquid/solid ratios of 8, 20, and 40:1, bracketing (or containing) the MLC for that replicate. For example, to calculate the AMC8, the quantity of a target constituent leached was determined over a liquid/solid ratio of 4 on each side of the MLC liquid/solid ratio. After the quantity leached in the appropriate control (over the same liquid/solid ratio) had been subtracted, this quantity, in milligrams, was then converted to an average concentration by dividing by the volume of leachate, in liters, collected over that liquid/solid ratio. If the MLC liquid/solid ratio was less than 4:1, then the quantity leached over the initial liquid/solid ratio of 8:1 was used. The same methodology was used to calculate AMC20 and AMC40, using liquid/solid ratios of 20 and 40:1, respectively. To calculate IAC8, the total quantity of the target constituent leached over the first eight liquid/solid ratios was determined, followed by subtracting the quantity leached in the control leachate at the same liquid/solid ratio and dividing the difference by the volume of leachate collected. Target concentrations for the 25 inorganic and organic constituents in terms of MLC, AMC8, AMC20, AMC40, and IAC8 are listed in Table 61.

Thirty-two laboratory extraction methods were ranked to best simulate the above five sets of target concentrations. The extraction methods, conducted in duplicate, consisted of an upflow-column and a rotary-batch procedure, using four media--(1) a 0.1 M sodium acetate pH 5 buffer, (2) carbonic acid (carbon-dioxide-saturated de-ionized distilled water), (3) de-ionized distilled water, and (4) MWL from the field lysimeter test facility (ORNL/MWL)--at four liquid/solid ratios--2.5, 5, 10, and 20:1. Two ancillary extractions were included: (1) the RCRA-approved EP and (2) a bi-sequential extraction procedure developed to extract high concentrations of acid soluble metals in predominantly alkaline wastes. Extractions using the two ancillary methods, however, were not replicated. Concentrations of the 25 target chemicals were determined in the laboratory extractions of the respective wastes and compared to the five sets of target concentrations.

The difference between the laboratory concentrations and their respective target concentrations was defined as follows:

$$\text{Difference} = [\text{abs}(TC - LC)/TC] \times 100,$$

where

TC is the target concentration,
LC is the laboratory concentration, and
abs( ) is the absolute value and the difference is expressed in percent.

An average difference between the target concentration and the laboratory concentration was then determined for each of the 32 treatments and each of the 25 target constituents. An overall average difference was then determined for each of the categories--(1) inorganic, (2) organic, and (3) inorganic and organic target constituents--for each of the five sets of target concentrations--MLC, AMC8, AMC20, AMC40, and IAC8. These average differences were then ranked from the lowest to the highest. For the 32 replicated extraction methods (all but the EP and the bi-sequential extraction methods were duplicated), significant differences between extractions could be determined in an analysis of variance testing procedure (Statistical Analysis System [SAS] software package [SAS Institute 1982]).

The best ranked extraction methods for estimating MLC target concentrations were those that used ORNL/MWL as an extraction medium. There appeared not to be a preference relative to the type of extraction procedure (upflow-column or rotary-batch) with ORNL/MWL; however, liquid/solid ratios at 10:1 and less ranked consistently better than 20:1 ratios. Of the top 10 ranked methods, sodium acetate and carbonic acid extraction media in rotary-batch extraction procedures at liquid/solid ratios of 2.5 to 10:1 were the only synthetic media. Sodium acetate at liquid/solid ratios of 5 and 10:1 ranked slightly better than carbonic acid at 2.5 and 5:1 liquid/solid ratios. Statistically, there were no significant differences between these sodium acetate and carbonic acid extractions, suggesting that the choice of any of the four extraction methods would be satisfactory.

Carbonic acid was the extracting medium in the five top ranked methods for estimating AMC8 target concentrations. These, however, were not statistically different from others among the first 26 ranked methods, largely because of the high variability between replicates in the extraction of organic compounds and the single degree of freedom inherent in the experimental design. Regardless of the statistical interpretations, it is quite evident that to simulate AMC8 target concentrations, for wastes containing both organic and inorganic constituents, the preferred extracting media would be carbonic acid. Because of the operational constraints of the upflow-column (e.g., inherently slow flow rates with wastes of low hydrologic conductivities) and the relatively small differences in ranking between the two procedures, the selection of the rotary-batch extraction procedure would be preferred over the upflow-column procedure. Choice of the specific liquid/solid ratio appeared not to be of major importance: the first five ranked methods (those using carbonic acid) included 5, 10, and 20:1 liquid/solid ratios.

For AMC target concentrations at higher liquid/solid ratios (20 and 40:1 as compared to 8:1) the preferred extraction method generally utilized extraction procedures at 20:1 liquid/solid ratios and less aggressive extracting media (i.e., de-ionized distilled water and carbonic acid rather than sodium acetate or ORNL/MWL). The best ranked extraction method for both AMC20 and AMC40 (inorganic and organic target constituents) was carbonic acid in an upflow-column at a 20:1 liquid/solid ratio. For AMC40, the rotary-batch procedure, using the same medium and liquid/solid ratio, was ranked 2; for AMC20 the procedure was ranked 4. Over all the AMC target sets (AMC8, AMC20, and AMC40), carbonic acid in a rotary-batch extraction procedure at a liquid/solid ratio of 20:1 ranked, respectively, 1, 4, and 5 for extracting inorganic and organic target constituents from the four industrial wastes.

The outstanding observation in the ranking of the extraction methods to simulate IAC8 target concentrations (inorganic and organic) was the total dominance of de-ionized distilled water as the extraction medium in the top five rankings. The major differences in the target concentrations for IAC8 and AMC8 were the lower IAC8 values for Ni and Zn in the electroplating waste (Table 61). In retrospect, the high rankings for de-ionized distilled water were not surprising in that the pH of the leachates from the two alkaline wastes (API/incinerator ash and electroplating waste) was relatively high during the first 8:1 liquid/solid leaching interval (pH values ranged from 9.5 to 8.1 and 8.4 to 6.4, respectively). Under these conditions, the interactions of the MWL on the wastes were predominantly the same as interactions of distilled water (i.e., it leached out the water-soluble constituents, but left the acid-soluble metals such as Ni and Zn until the leachate pH became lower later in the leaching).

The objective of the research was to develop a laboratory extraction method for solid wastes that simulated the concentrations of potentially hazardous inorganic and organic constituents in leachates that result from co-disposing industrial wastes with municipal wastes in landfills. The intent of the research was to produce a scientific rationale and data base supporting the selection of such an extraction method. The intended characteristics of the extraction procedure included the following:

1. Ability to simulate leaching in a landfill containing

municipal and industrial wastes in proportions of about 95% and 5% by weight, respectively.

2. Compatibility with biological toxicity tests (mutagenic, aquatic, and phytotoxic).

3. Low cost (relatively inexpensive to conduct in terms of time, equipment, and personnel).

From this research and previous research, it appears that no single extraction method will be optimal, inclusive of all wastes, waste constituents, or landfill scenarios. This research has demonstrated the relative effectiveness of a number of extraction methods for a variety of wastes and target conditions. The final section of any one method or combination of methods will depend on what leachate target concentrations are to be simulated. The data presented here do suggest that the use of carbonic acid as an extracting medium in a rotary-batch procedure be thoroughly considered because such an extraction method would fulfill many of the above criteria. Compatibility of carbonic acid extractions with numerous biotesting protocol should add significantly in evaluating the toxicity of solid waste leachates.

FUTURE RESEARCH

To expand the existing data base and to verify the selection of EP-III, 11 additional wastes are being leached in the lysimeter test facility. These wastes include contaminated soil (containing Cd, Hg, and PCBs), ammonia lime still bottoms (containing phenols, PAHs, volatile organic compounds, ammonia, and inorganic elements), a variety of oily sludges (containing volatile organic compounds and PAHs), a fly ash, and resource recovery ashes from four waste-to-energy facilities (all containing leachable inorganic constituents). Leaching characteristics of these wastes by MWL are being determined. Laboratory extractions will be conducted using the method selected as EP-III to evaluate how closely it predicts lysimeter leachate concentrations. To assess the accuracy and variability of EP-III between laboratories, selected wastes will be used with EP-III in multi-laboratory comparisons.

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REFERENCES

Brown, D. K., M. P. Maskarinec, F. W. Larimer, and C. W. Francis. 1983. Mobility of Organic Compounds from Hazardous Waste. EPA-600/S4-83-001. Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Las Vegas.

Chian, E. S. K., and F. B. DeWalle. 1977. Evaluation of Leachate Treatment, Volume 1. Characterization of Leachate. EPA-600/2-77-186a. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

Daniel, C., and F. S. Wood. 1980. Fitting Equations to Data: Computer Analysis of Multifactor Data. John Wiley and Sons, New York.

Dyer, F., J. Emery, and L. Lendicotte. 1962. A Comprehensive Study of the Neutron Activation Analysis of Uranium by Delayed Neutron Counting. ORNL-3342. Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Epler, J. L., et al. 1980. Toxicity of Leachates. EPA-600/2-80-057. Office of Research and Development, U.S. Environmental Protection Agency, Washington, D.C. 134 pp.

Feldman, C. 1974. Perchloric Acid Procedure for Wet-Ashing Organics for the Determination of Mercury (and Other Metals). Anal. Chem. 46:1606-1609.

Feldman, C. 1979. Improvements in the Arsine Accumulation-Helium Glow Detector Procedures for Determining Traces of Arsenic. Anal. Chem. 51:664-669.

Fuller, W. H., B. A. Alesii, and G. E. Carter. 1979. Behavior of Municipal Solid Waste Leachate. I. Composition Variations. J. Environ. Sci. Health A14(6):461-485.

Fungaroli, A. A., and R. L. Steiner. 1979. Investigation of Sanitary Landfill Behavior, Volume 1. Final Report, EPA-600/2-79-053a. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

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Griest, W. H., M. P. Maskarinec, and B. A. Tomkins. 1983. Chemical and Physical Characterization and Management of Feed/Process/Product/Effluent Samples from H-Coal Plant. ORNL/TM-8911. Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Ham, R. K., M. A. Anderson, R. Stanforth, and R. Stegmann. 1978. The Development of a Leaching Test for Industrial Wastes. pp. 33-46. IN Land Disposal of Hazardous Wastes. Proc. of Fourth Annual Research Symposium. EPA-600/9-78-016. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

Ham, R. K., M. A. Anderson, R. Stegmann, and R. Stanforth. 1979a. Background Study on the Development of a Standard Leaching Test. EPA-600/2-79-109. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

Ham, R. K., M. A. Anderson, R. Stegmann, and R. Stanforth. 1979b. Comparison of Trace Waste Leaching Tests. EPA-600/2-79-071. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

Hagerty, D. J., J. L. Pavoni, and J. E. Herr, Jr. 1973. Solid Waste Management. Chapter 10. IN Sanitary Landfill. Van Nostrand Reinhold Co.

Ho, C. H., B. R. Clark, and M. R. Guerin. 1976. Direct Analysis of Organic Compounds in Aqueous By-Products from Fossil Fuel Conversion Processes: Oil Shale Retorting, Synthane Coal Gasification, and COED Coal Liquefaction. J. Environ. Sci. Health All(7):481-489.

Johansen, O. J., and D. A. Carlson. 1976. Characterization of Sanitary Landfill Leachates. Water Res. 10:1129-1134.

Kinman, R. H., and J. J. Walsh. 1980. Leachate from Municipal and Industrial Waste Landfill Simulators. pp. 203-221. IN Disposal of Hazardous Waste (D. Black, Ed.). Proc. of the Sixth Annual Research Symposium. EPA-600/9-80-010. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

Kinman, R. N., J. I. Rickabaugh, and J. J. Walsh. 1982. Leachate from Co-Disposal of Municipal and Hazardous Waste in Landfill Simulators. pp. 274-293. In Land Disposal: Municipal Solid Waste. Proc. of Eighth Annual Research Symposium (in press).

Kmet, P., and P. M. McGinley. 1982. Chemical Characteristics of Leachate From Municipal Solid Waste Landfills in Wisconsin. IN Proc., Fifth Annual Madison Conference of Applied Research and Practice on Municipal and Industrial Waste. Sept. 22-24, 1982. Department of Engineering and Applied Science, University of Wisconsin, Madison, Wisconsin.

Lowenbach, W. 1978. Compilation and Evaluation of Leaching Test Methods. EPA/600-2-78-095, Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio.

Manning, D. L., and M. P. Maskarinec. 1983. Determination of Low Molecular Weight Carboxylic Acids in Water by HPLC with Conductivity Detection. J. Liq. Chromatogr. 6(4):705-714.

Meyers, T. E., N. R. Francingues, D. W. Thompson, and P. G. Malme. 1980. Chemical Stabilized Industrial Wastes in a Sanitary Landfill Environment. pp. 223-241. IN Disposal of Hazardous Waste (D. Black, Ed.). Proc., Sixth Annual Research Symposium. EPA-600/9-80-010. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

Millemann, R. E., B. R. Parkhurst, and N. T. Edwards. 1981. Toxicity to Daphnia magna and Terrestrial Plants of Solid Waste Leachates from Coal Conversion Processes. pp. 237-247. IN Proc., Twentieth Hanford Life Sciences Symposium on Coal Conversion and the Environment. Battelle Pacific Northwest Laboratory, Richland, Washington.

Qasin, S. R., and J. C. Burchinal. 1976. Leaching from Simulated Landfills. J. Water Pollut. Control Fed. 42(3):371-379.

Regan, R. W. 1983. Effect of Filtration Protocol on Polycyclic Aromatic Hydrocarbon Recovery from Industrial Sludges. Faculty Research Participation Program. Summary Report to Oak Ridge Associated Universities, Oak Ridge, Tennessee.

SAS Institute, Inc. 1982. SAS Users Guide Statistics, 1982 Edition. Cary, North Carolina. 594 pp.

Schuetzle, D., T. J. Prater, and S. R. Roddell. 1975. Sampling and Analysis of Emissions from Stationary Sources. I. Odor and Total Hydrocarbons. J. Air Pollut. Control Fed. 24(9):925-932.

Sheffer, F., R. Keckuth, and H. Lorenz. 1965. A New Method for the Separation of Organic Acids on DEAE-Sephadex A-25. Naturwissenschaften 52:517.

Strang, D. R. 1977. The Effects of Industrial Sludges on Landfill Leachates and Gas. pp. 41-54. IN Management of Gas and Leachate in Landfills. Proc., Third Annual Municipal Solid Waste Research Symposium. EPA-600/9-77-026. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

Swartzbaugh, J. T., R. L. Hentrich, Jr., and G. V. Sabel. 1978. Co-Disposal of Industrial and Municipal Waste in a Landfill. pp. 129-151. IN Land Disposal of Hazardous Wastes. Proc., Fourth Annual Research Symposium. EPA-600/9-78-016. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

Talmi, Y., and A. W. Andren. 1974. Determination of Selenium in Environmental Samples Using Gas Chromatography with a Microwave Emission Spectrometric Detection System. Anal. Chem. 46:2122-2126.

U.S. Environmental Protection Agency (USEPA). 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-010. Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati. 460 pp.

U.S. Environmental Protection Agency (USEPA). 1980a. Identification and Listing of Hazardous Waste. In Environmental Protection Agency Hazardous Waste Management System. 40 CFR 261.24.

U.S. Environmental Protection Agency (USEPA). 1980b. Master Scheme for the Analysis of Organic Compounds in Water. USEPA, Research Triangle Park, North Carolina.

U.S. Environmental Protection Agency (USEPA). 1982. Test Methods for Evaluating Solid Wastes. Physical/Chemical Methods. SVV-846. Second Edition, Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Washington, D.C. 20460.

Walsh, J. J., and R. N. Kinman. 1979. Leachate and Gas Production Under Controlled Moisture Conditions. pp. 41-57. IN Land Disposal of Solid Wastes. Proc., the Fifth Annual Research Symposium. EPA-600/9-70. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio.

Walsh, J. J., R. N. Kinman, and J. I. Rickabaugh. 1981a. Leachate and Gas From Municipal Solid Waste Landfill Simulators. pp. 67-93. IN Land Disposal: Municipal Solid Waste. Proc., Seventh Annual Research Symposium. EPA-600/9-81-002a. Municipal Environmental Research Laboratory, U.S. Environmental Protection Agency, Cincinnati.

Walsh, J., G. Vogt, R. Kinman, and J. Rickabaugh. 1981b. Leachate and Gas from Municipal Solid Waste Landfill Simulators. pp. 84-105. IN Proc., Fourth Annual Madison Conference of Applied Research and Practice on Municipal and Industrial Waste. Department of Engineering and Applied Science, University of Wisconsin, Madison, Wisconsin.

Walsh, J., G. Vogt, R. Kinman, and J. Rickabaugh. 1981c. Demonstration of Landfill Gas Enhancement Techniques in Landfill Simulators. pp. 278-307. IN Proc., Fourth Annual Madison Conference of Applied Research and Practice on Municipal and Industrial Waste. Department of Engineering and Applied Science, University of Wisconsin, Madison, Wisconsin.

APPENDIX A

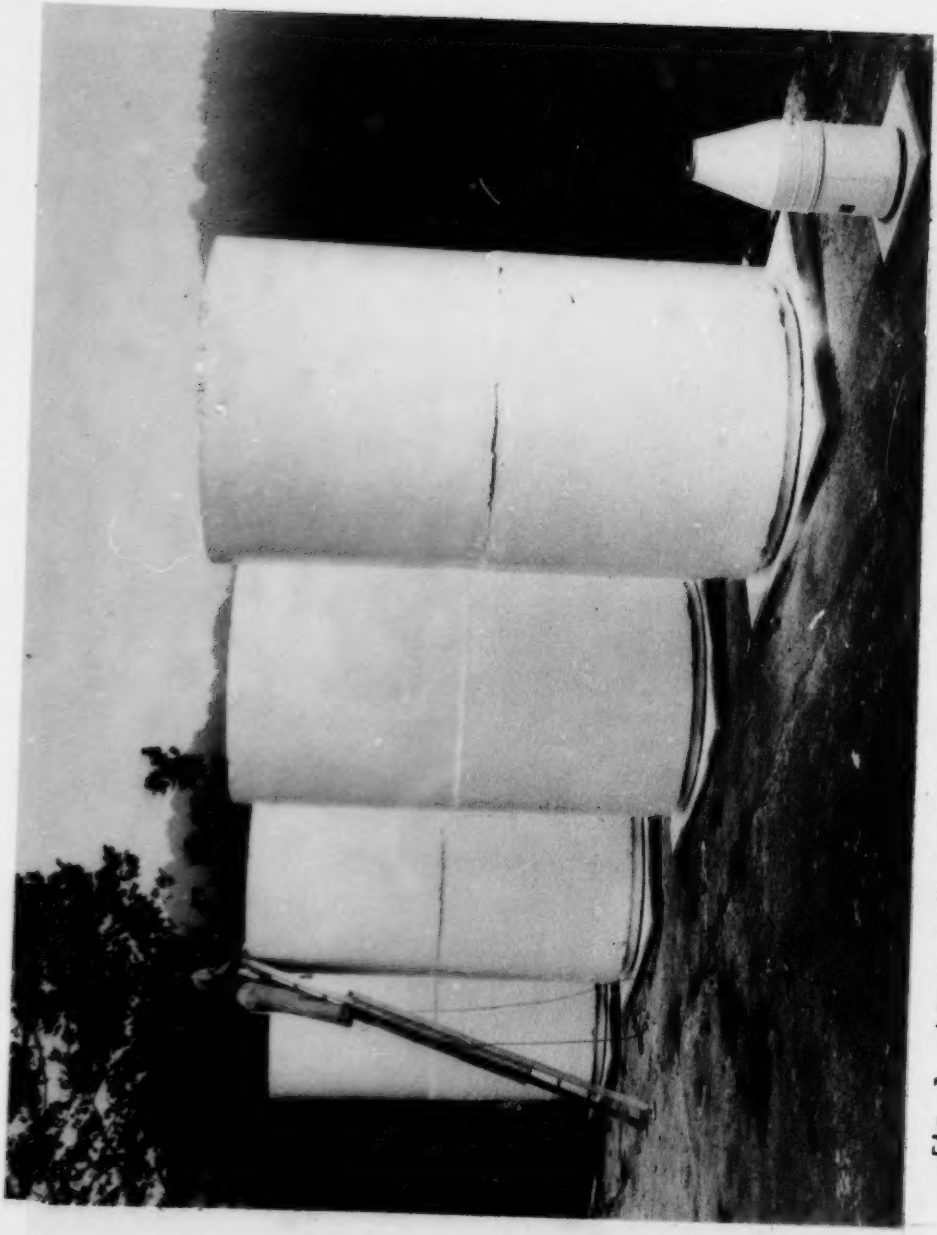

Fig. 1. Large-scale lysimeters used for generating municipal waste leachate.

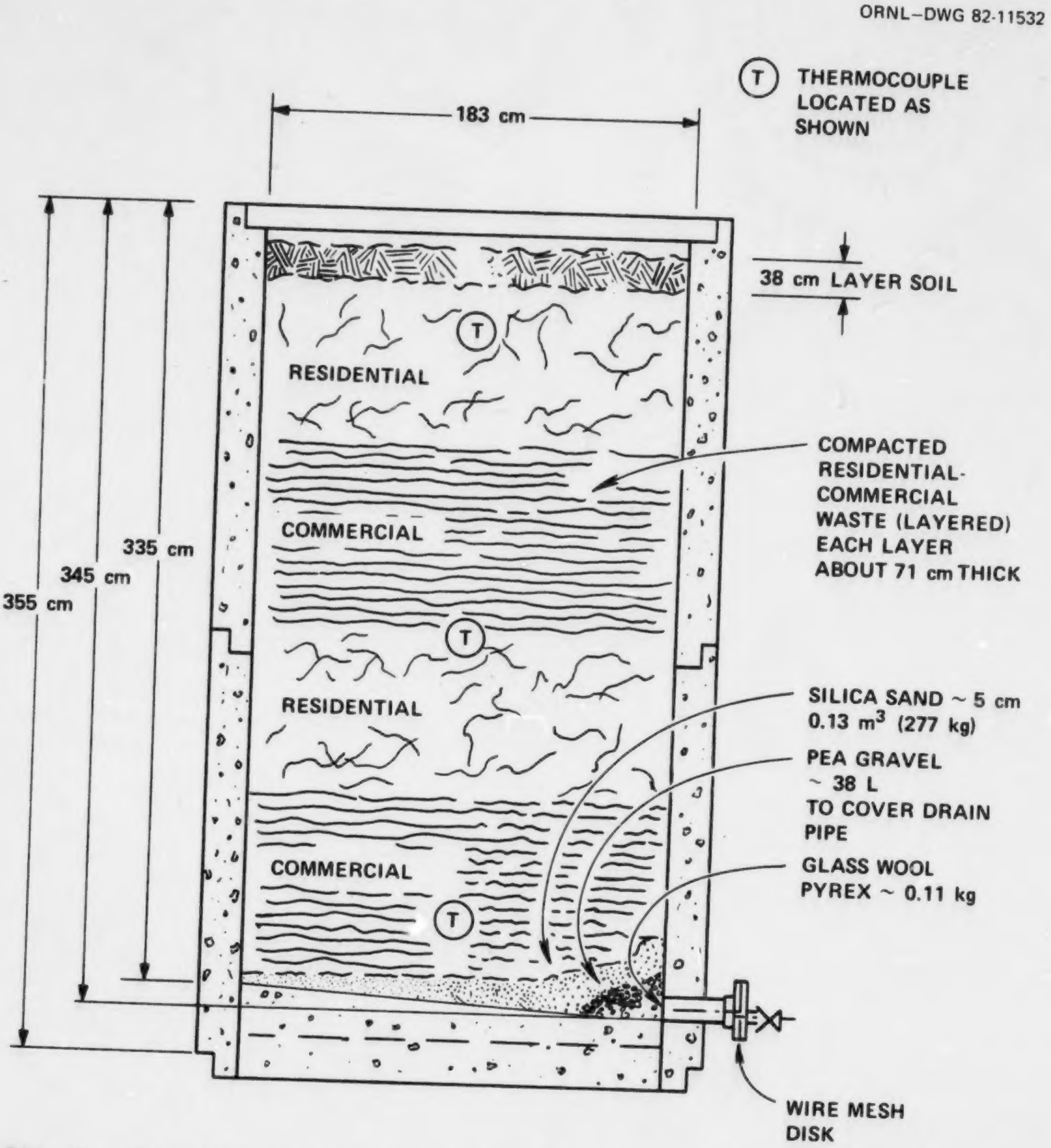
ORNL–DWG 82-11532
(T) THERMOCOUPLE LOCATED AS SHOWN
183 cm
38 cm LAYER SOIL
RESIDENTIAL
COMMERCIAL
RESIDENTIAL
COMMERCIAL
COMPACTED RESIDENTIAL-COMMERCIAL WASTE (LAYERED) EACH LAYER ABOUT 71 cm THICK
335 cm
345 cm
355 cm
SILICA SAND ~ 5 cm 0.13 m³ (277 kg)
PEA GRAVEL ~ 38 L TO COVER DRAIN PIPE
GLASS WOOL PYREX ~ 0.11 kg
WIRE MESH DISK

Fig. 2. Schematic of lysimeter containing residential and commercial wastes.

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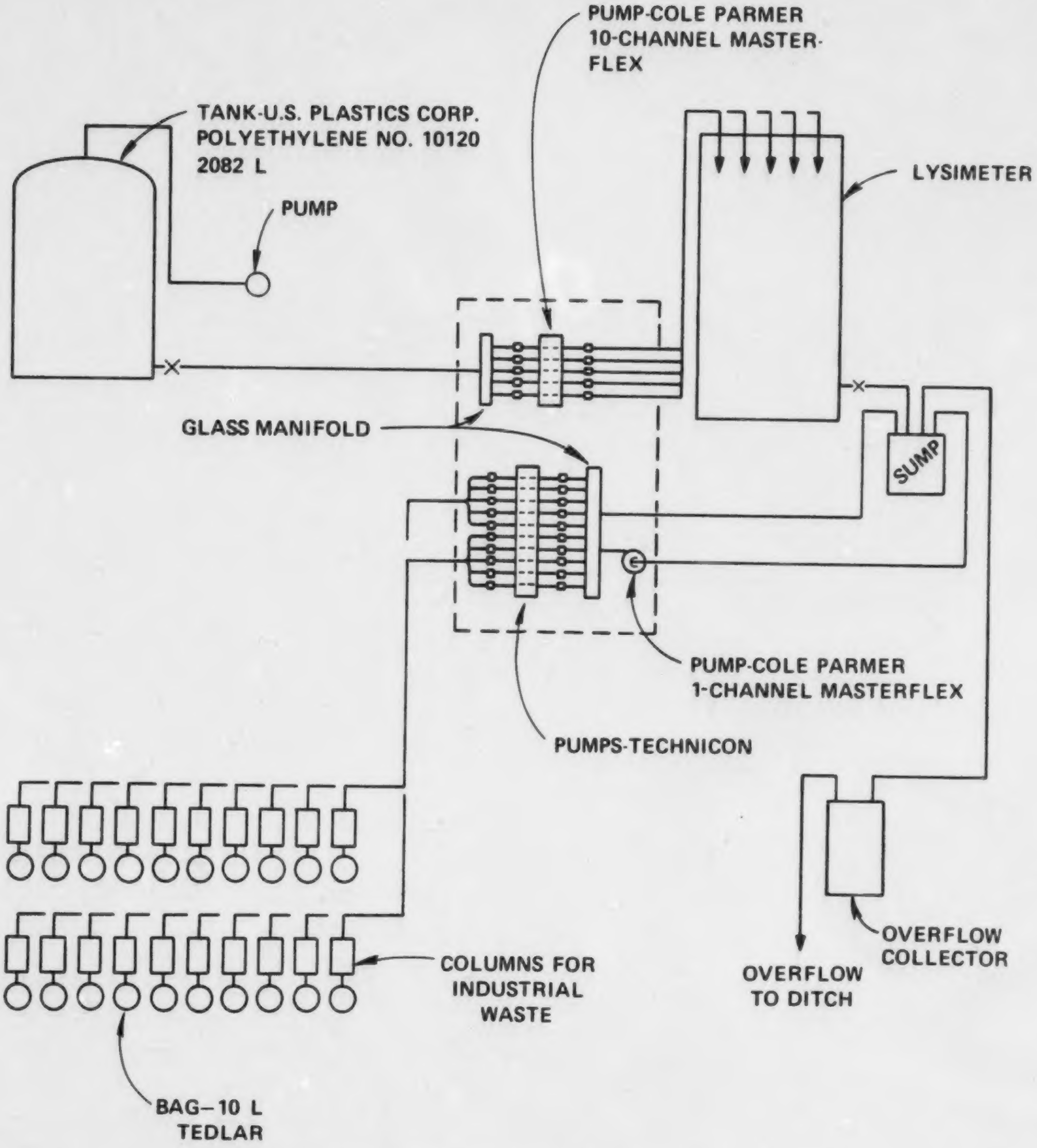
PUMP-COLE PARMER
10-CHANNEL MASTER-FLEX
TANK-U.S. PLASTICS CORP.
POLYETHYLENE NO. 10120
2082 L
PUMP
LYSIMETER
GLASS MANIFOLD
SUMP
PUMP-COLE PARMER
1-CHANNEL MASTERFLEX
PUMPS-TECHNICON
OVERFLOW
COLLECTOR
OVERFLOW
TO DITCH
COLUMNS FOR
INDUSTRIAL
WASTE
BAG–10 L
TEDLAR

Fig. 3. Flow diagram for lysimeter study.

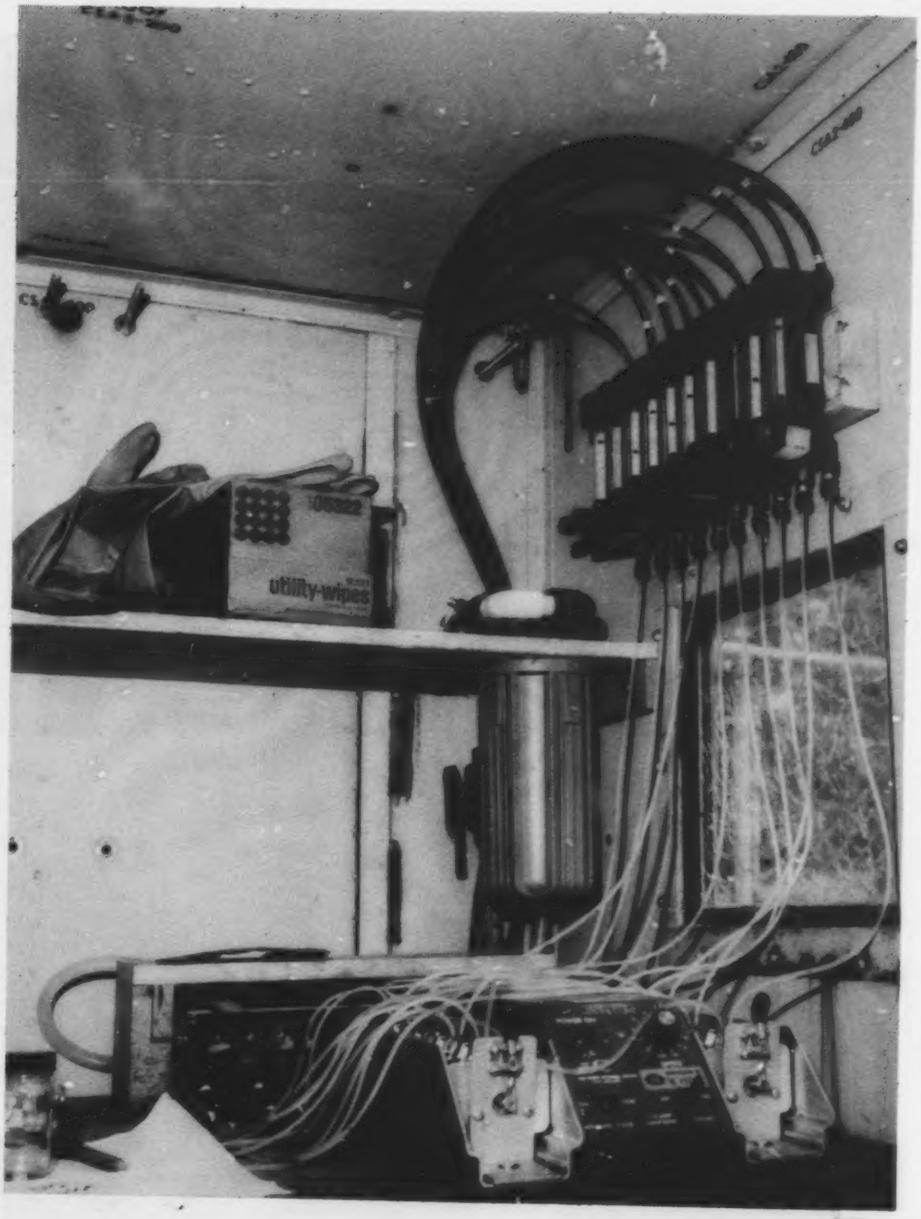
utility-wipes

Fig. 4. Pumps and flow meters used for delivery of water to lysimeters.

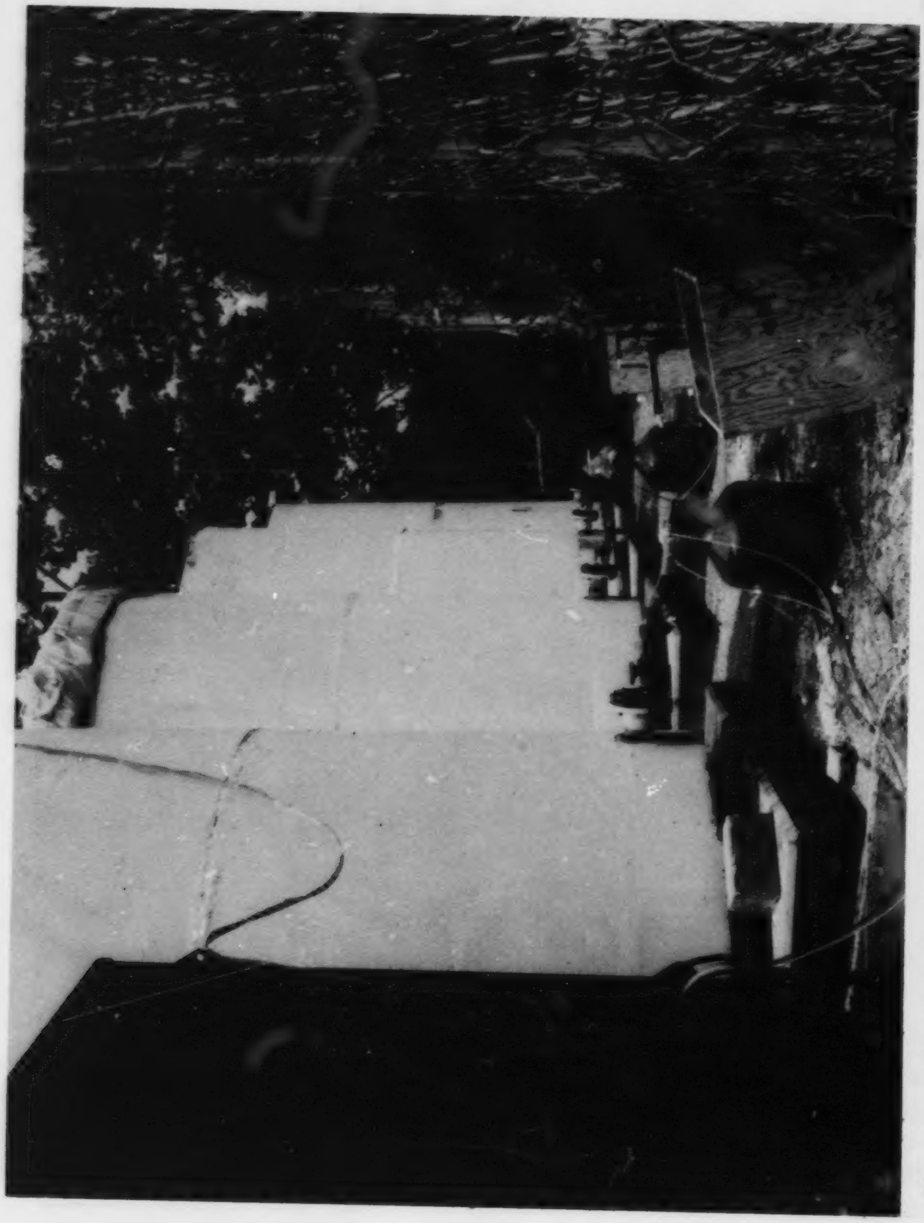

Fig. 5. View of the back side of the lysimeters and the 30-L carboys that served as sumps for the pumping of leachate to the industrial waste columns.

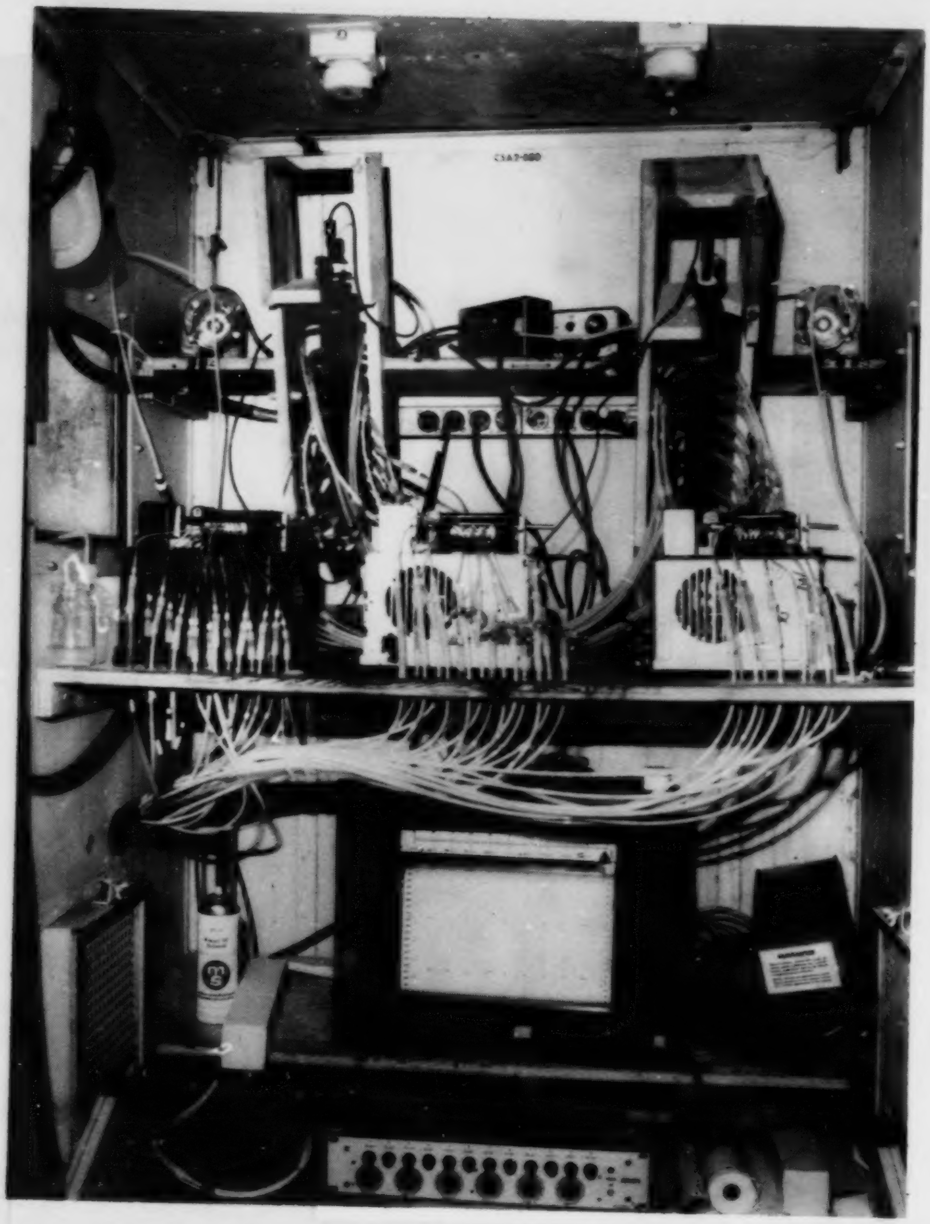

Fig. 6. Glass manifolds supporting the appropriate electrodes for monitoring pH and Eh of the municipal waste leachate.

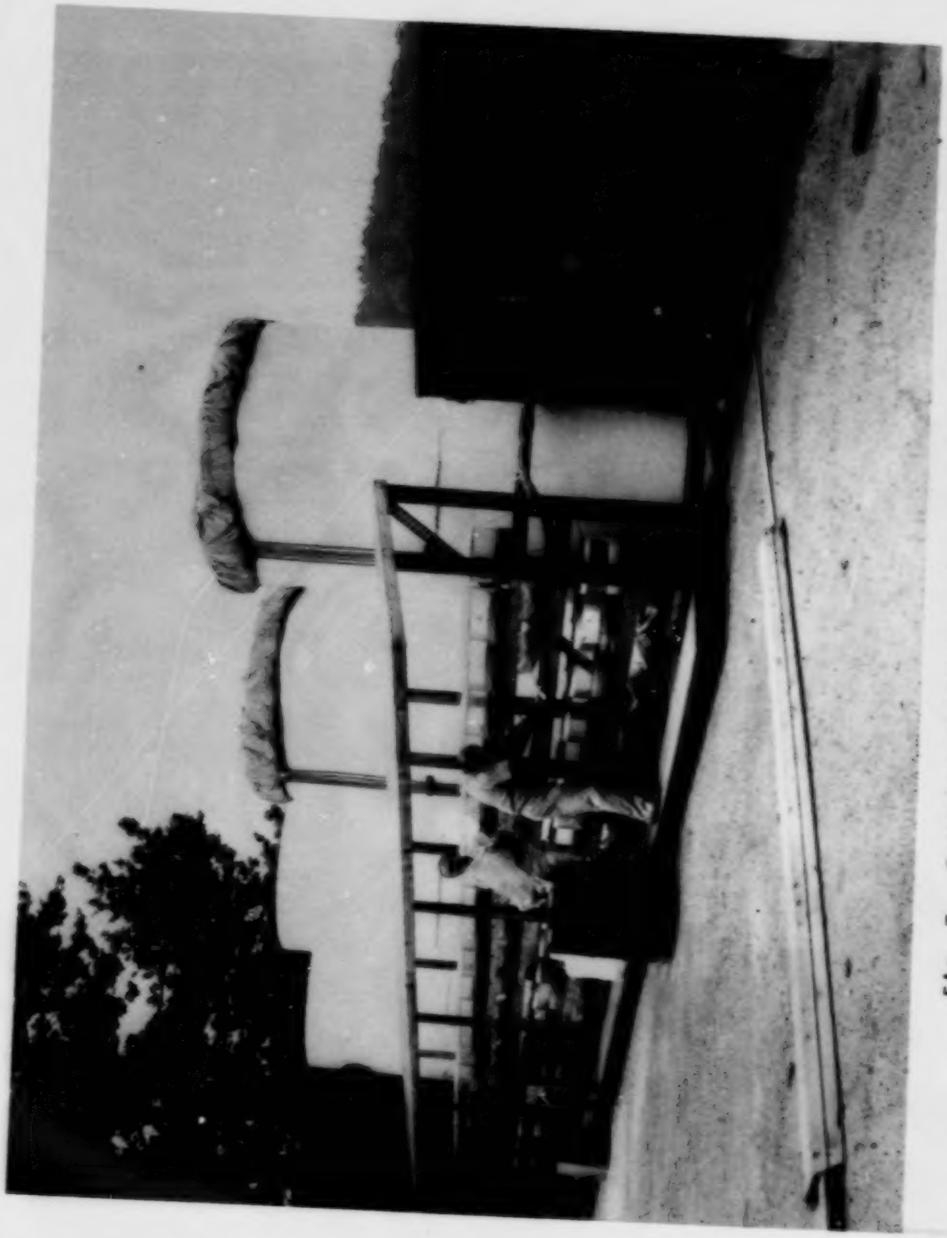

Fig. 7. Overview of field lysimeter test facility.

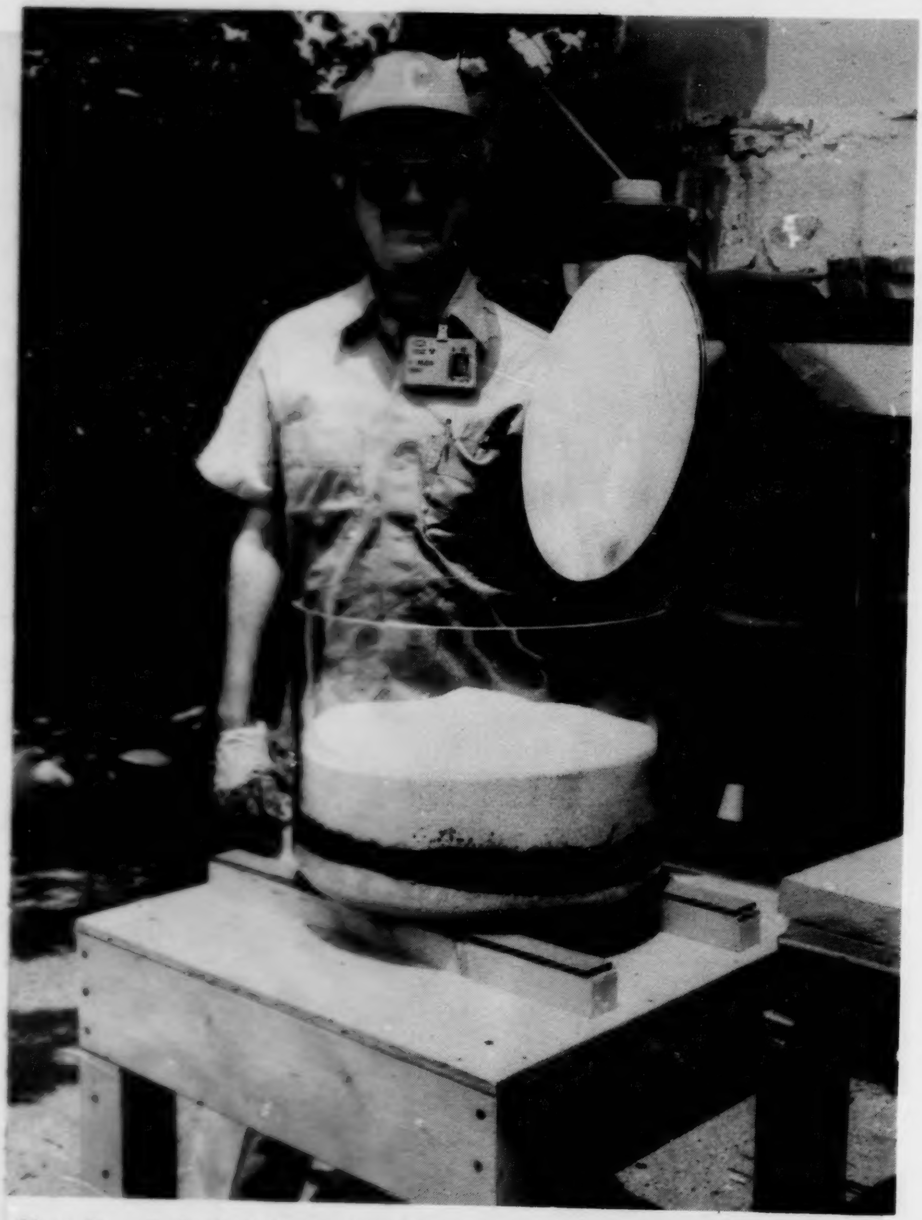

Fig. 8. Industrial wastes layered between layers of sand to distribute municipal waste leachate and reduce entrainment of waste particles by the leachate.

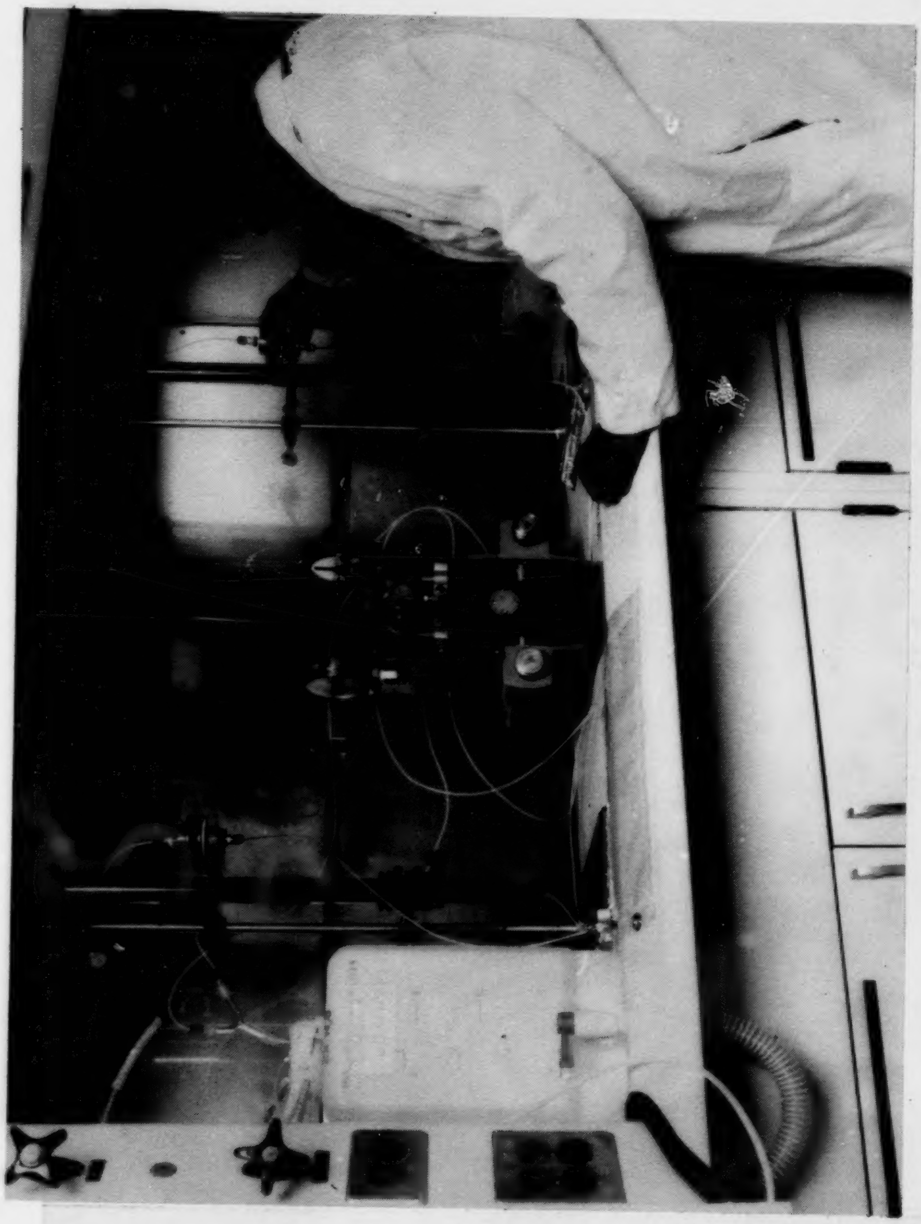

Fig. 9. Upflow columns and in-line filters used to extract industrial wastes.

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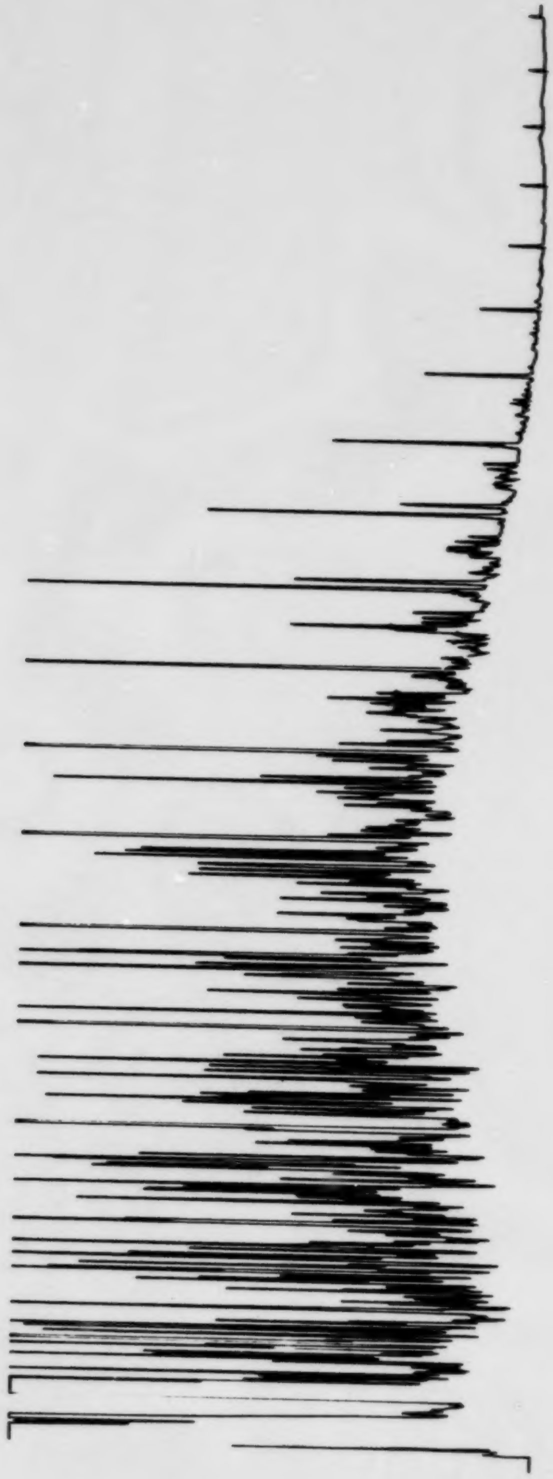

Fig. 10. Chromatogram of API/incinerator ash extract.

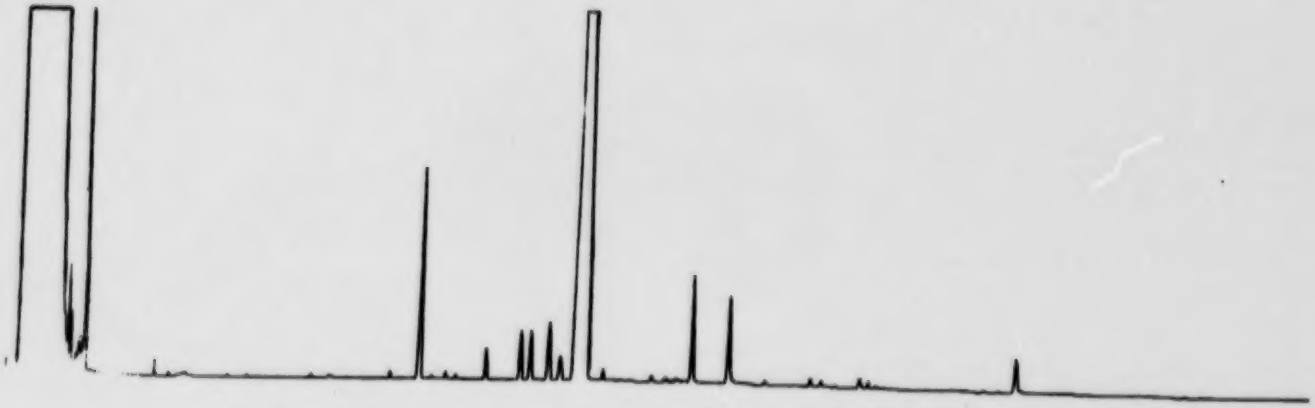

Fig. 11. Chromatogram of dichloroethylene still bottoms extract.

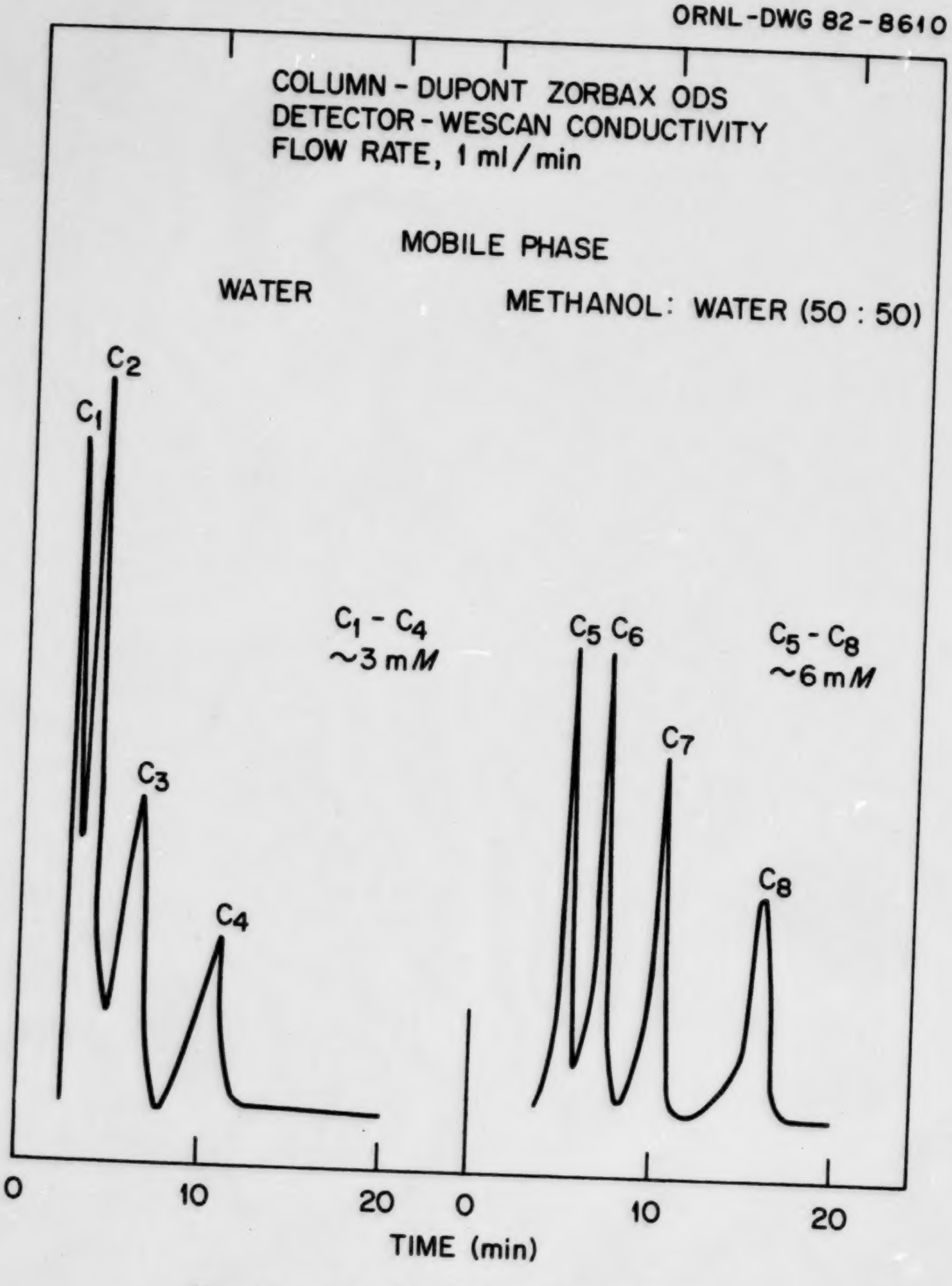

Fig. 12. Separation of carboxylic acids.

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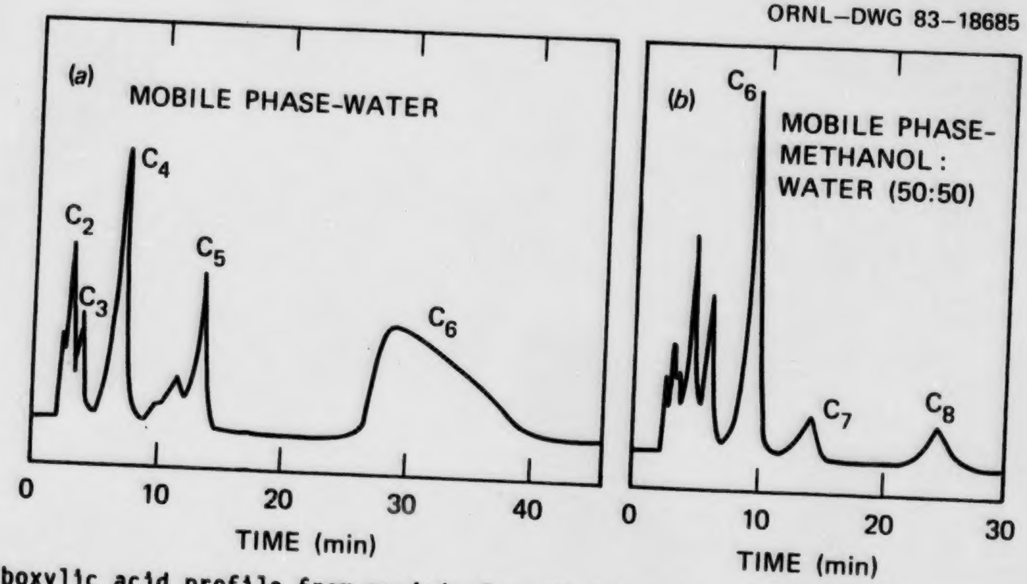

Fig. 13. Carboxylic acid profile from municipal waste leachate. Mobile phase flow rate = 1 mL/min.

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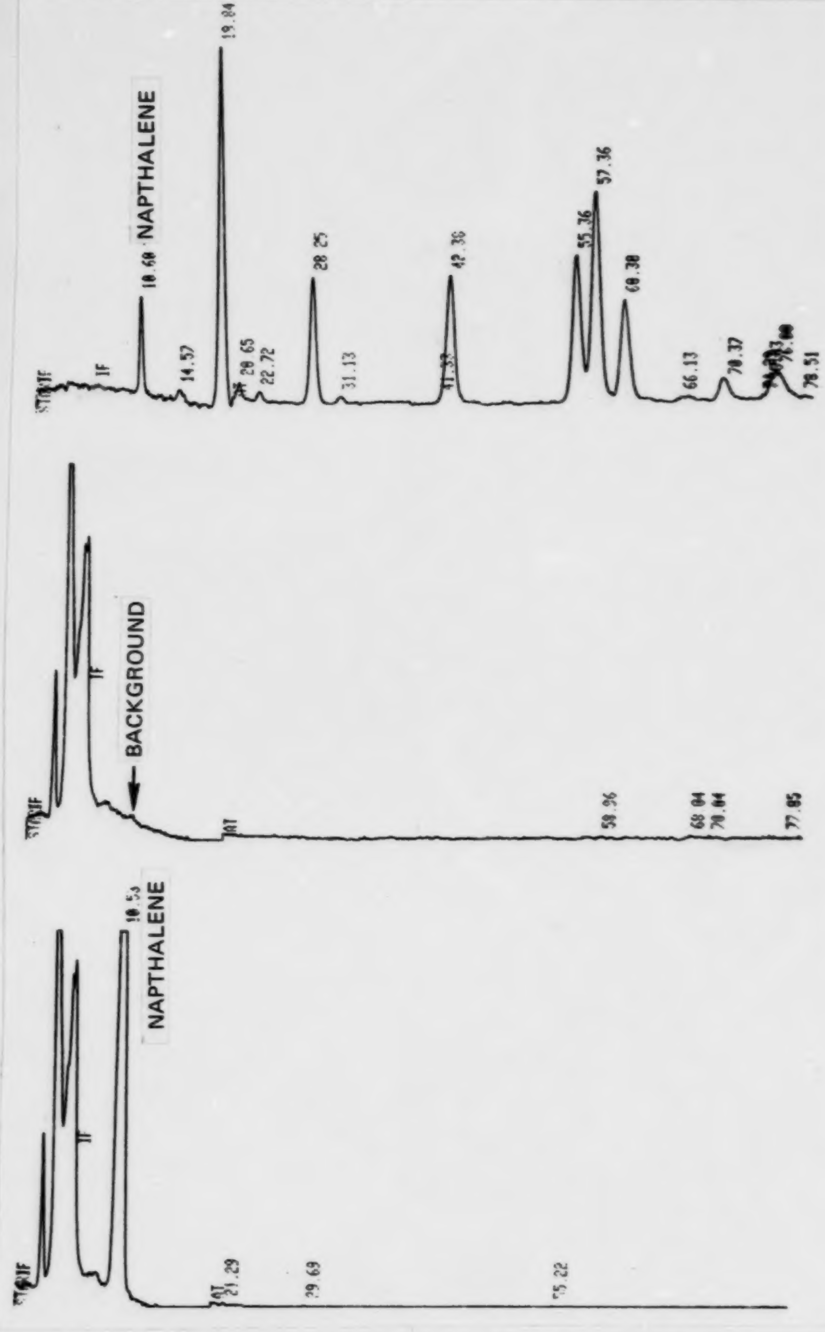

Fig. 14. Naphthalene standard, municipal waste leachate, and municipal waste leachate with added naphthalene.

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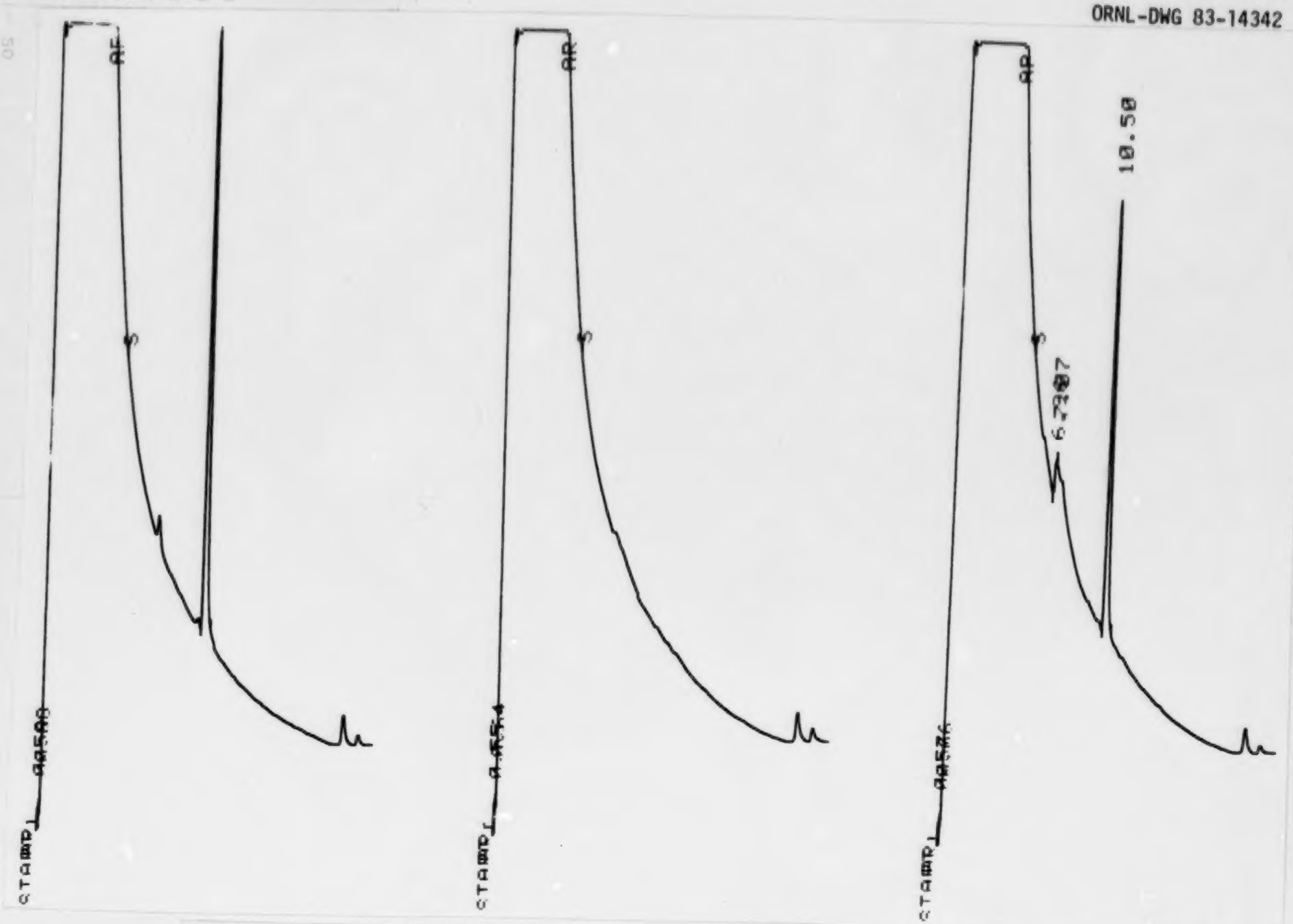

Fig. 15. Chromatograms of hexachlorobutadiene, municipal waste leachate, and municipal leachate with added hexachlorobutadiene.

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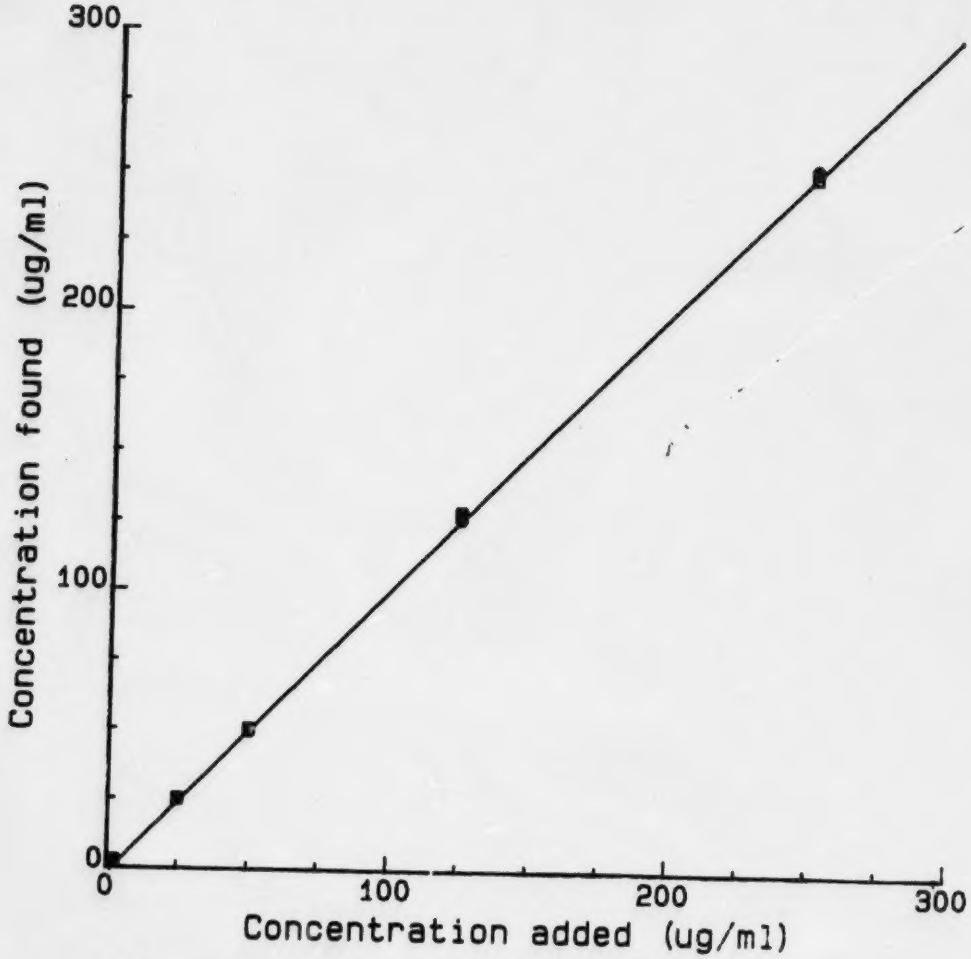

Fig. 16. Linearity of hexachlorobutadiene analysis.

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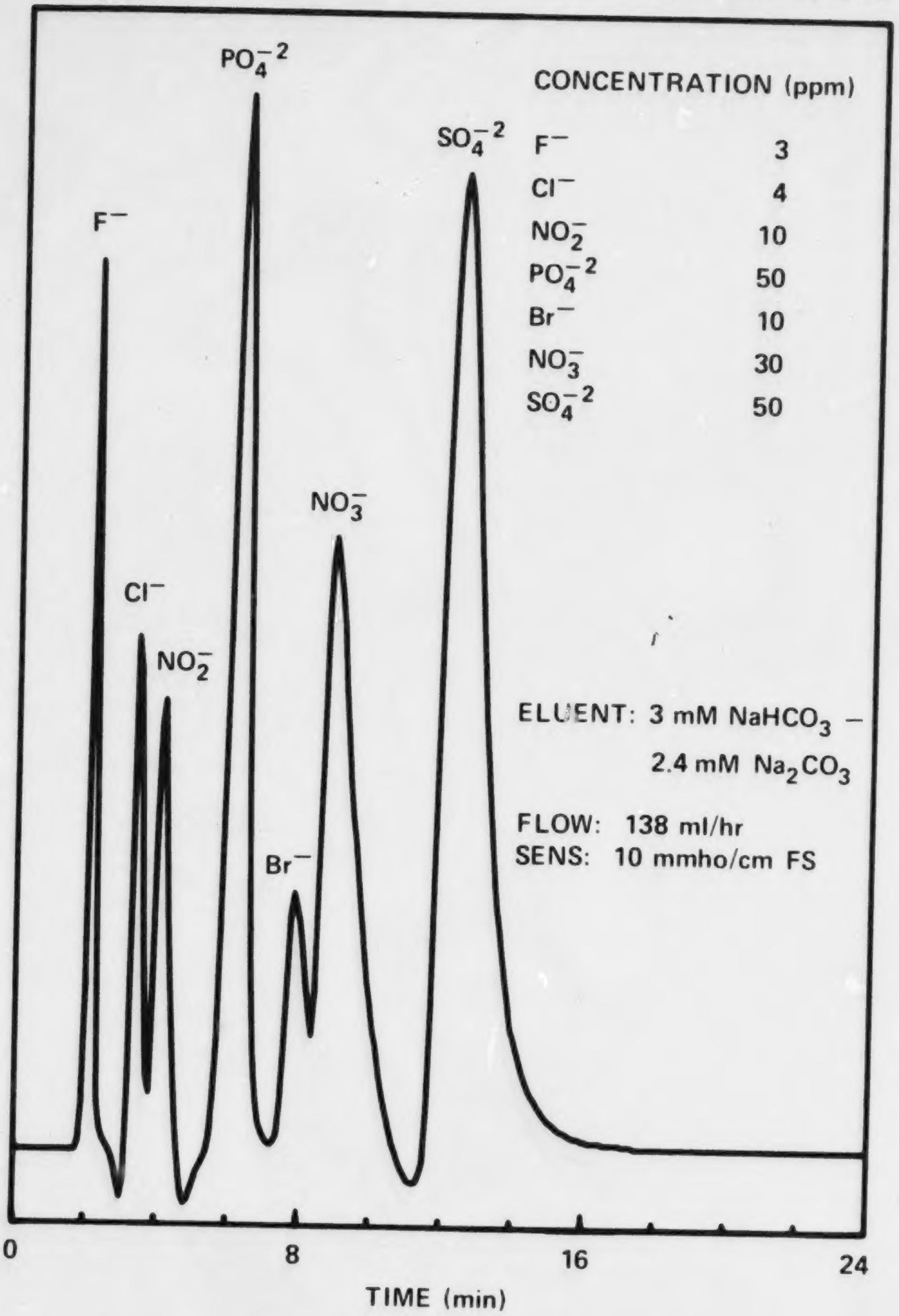

Fig. 17. Separation of anions by ion chromatography.

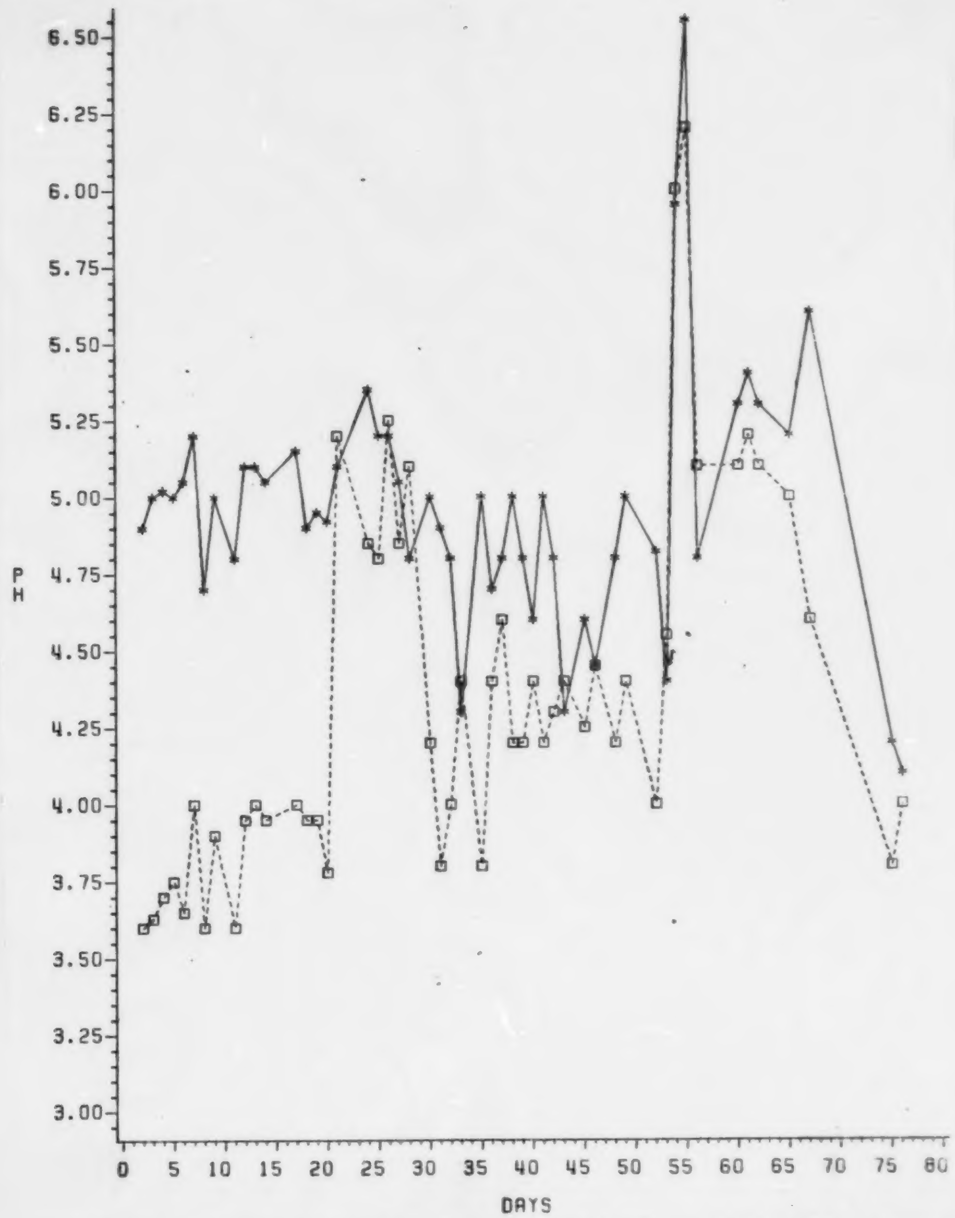

Fig. 18. The change in pH of the municipal waste leachate from lysimeters A and B during the 79 d of leaching.

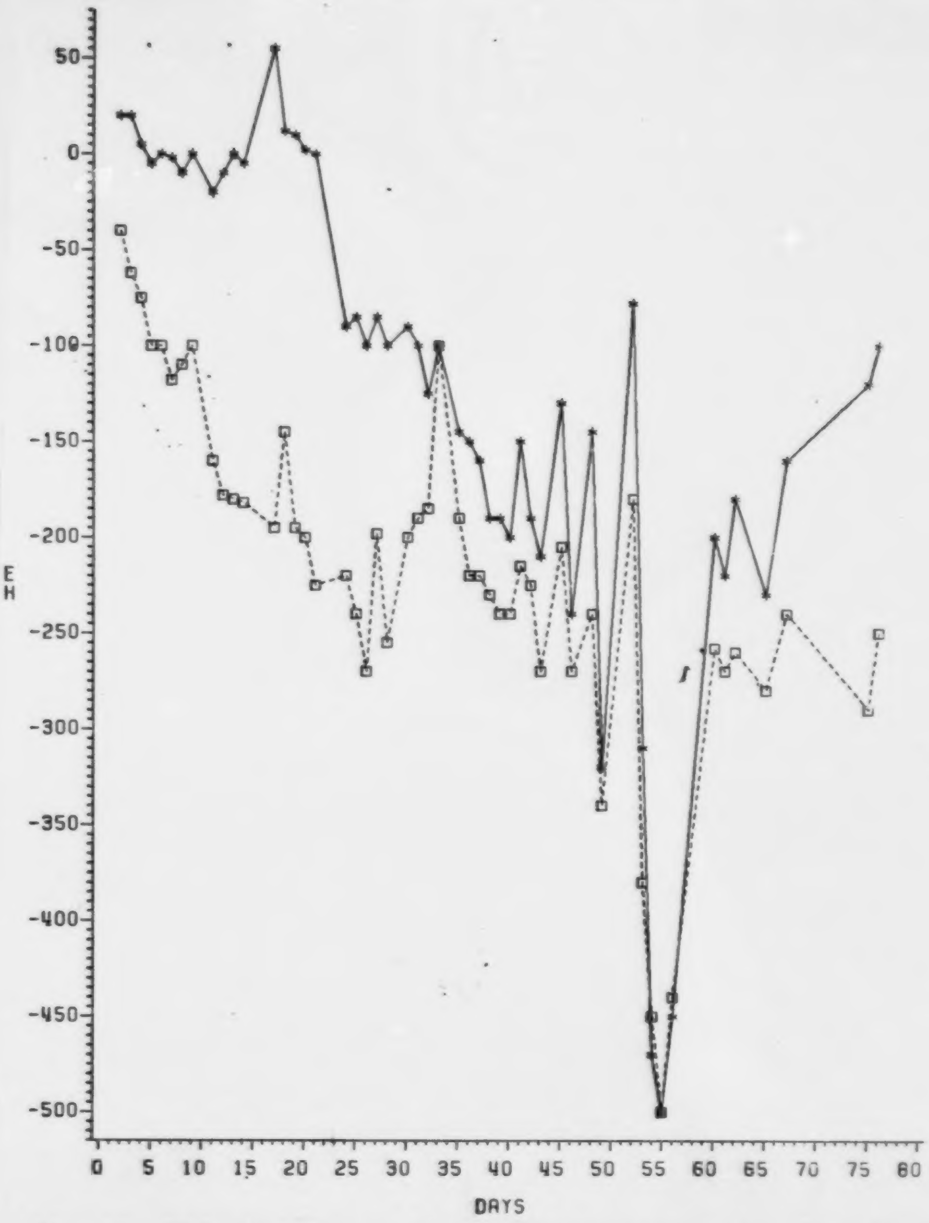

Fig. 19. The change in Eh of the municipal waste leachate from lysimeters A and B during the 79 d of leaching.

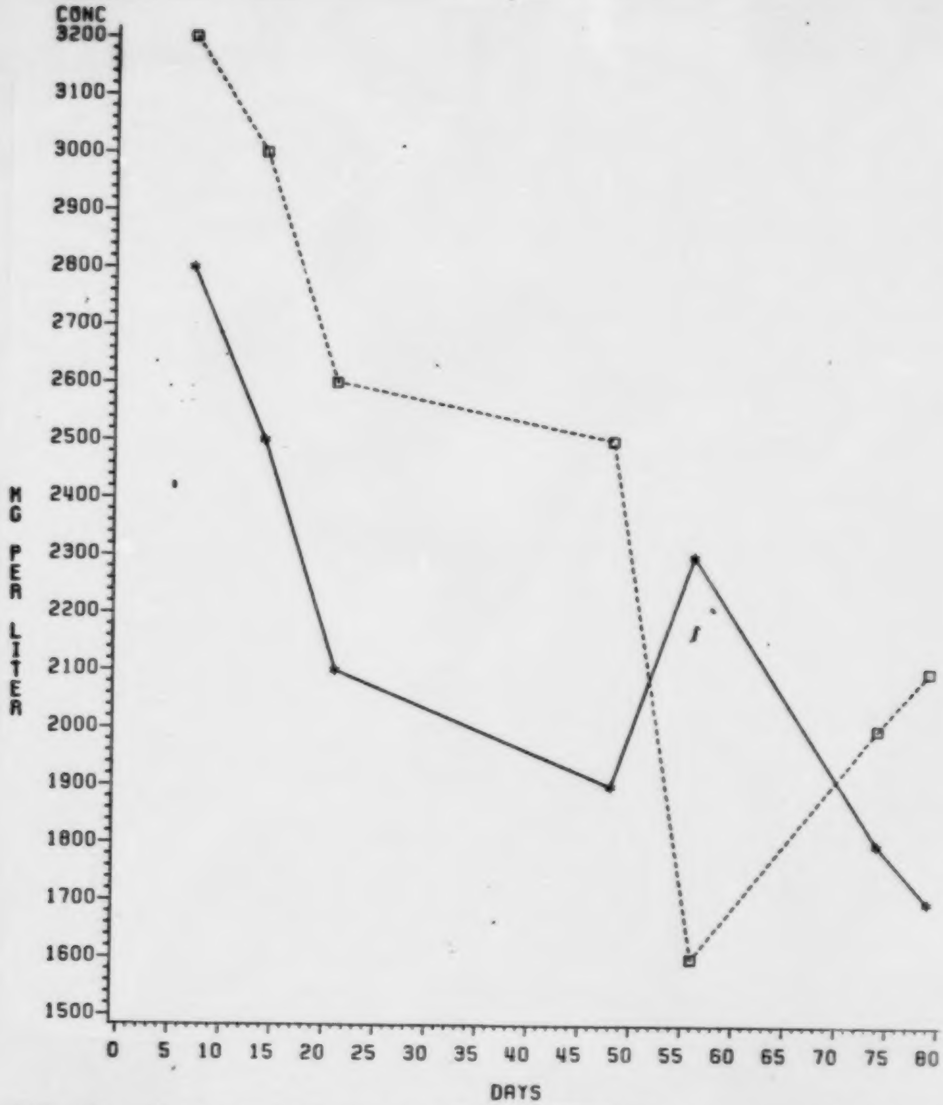

Fig. 20. Concentrations of total organic carbon (TOC) in municipal waste leachate from lysimeters A and B during the 79 d of leaching.

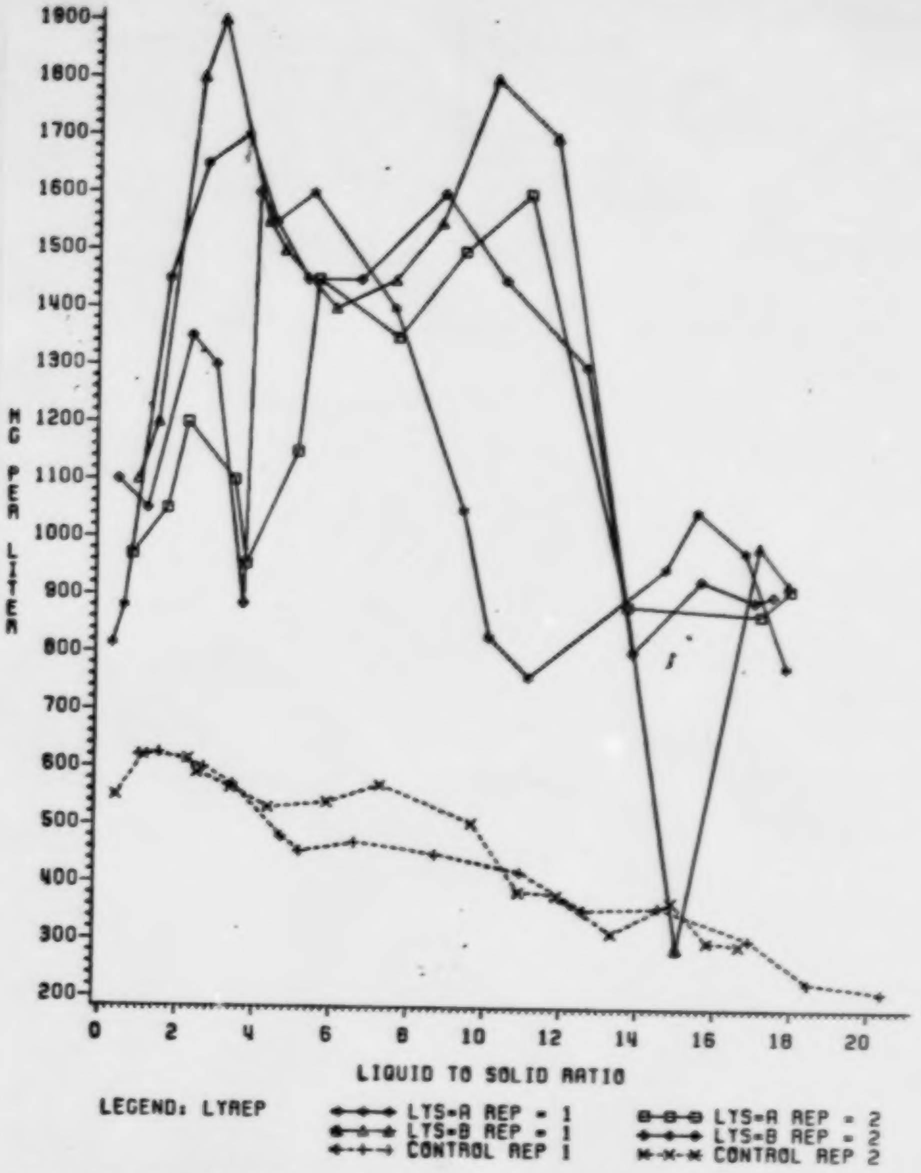

Fig. 21. Leaching of calcium from API/incinerator ash-Target.

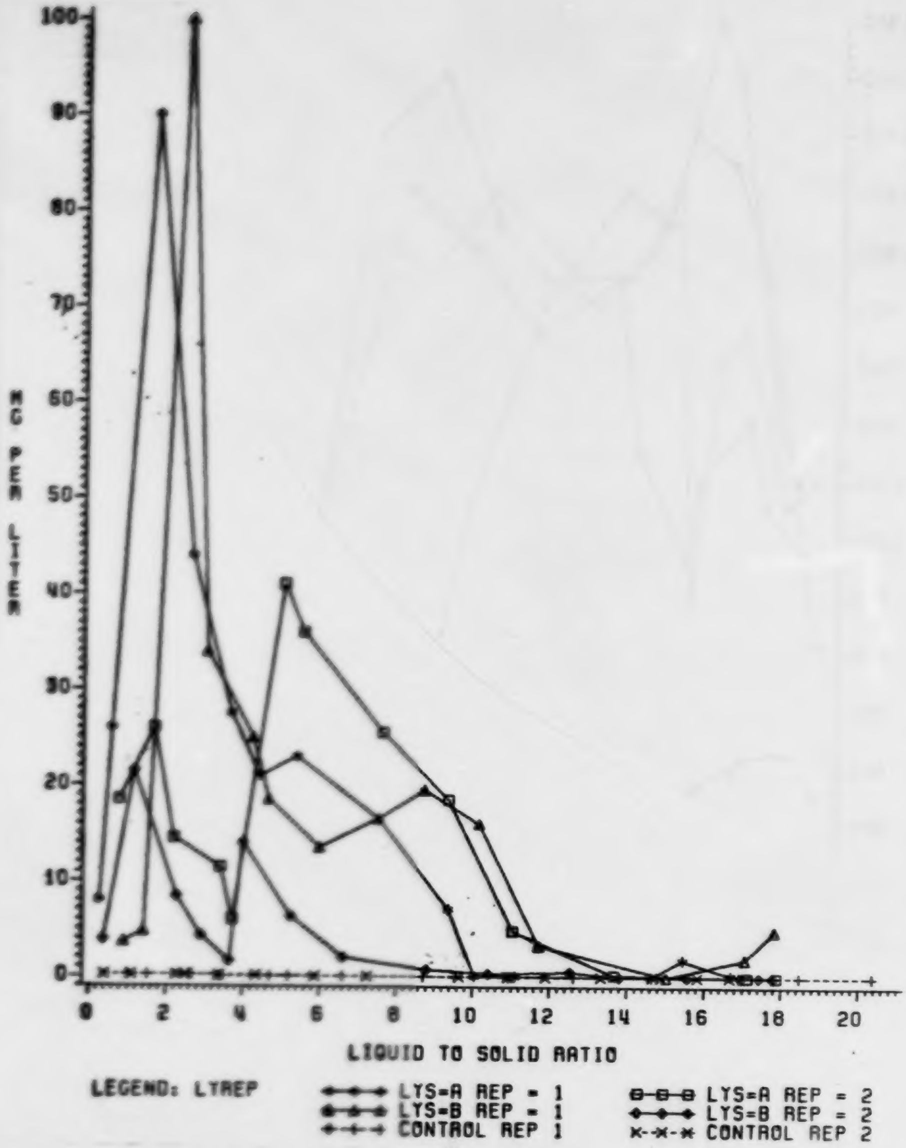

Fig. 22. Leaching of chromium from API/incinerator ash-Target.

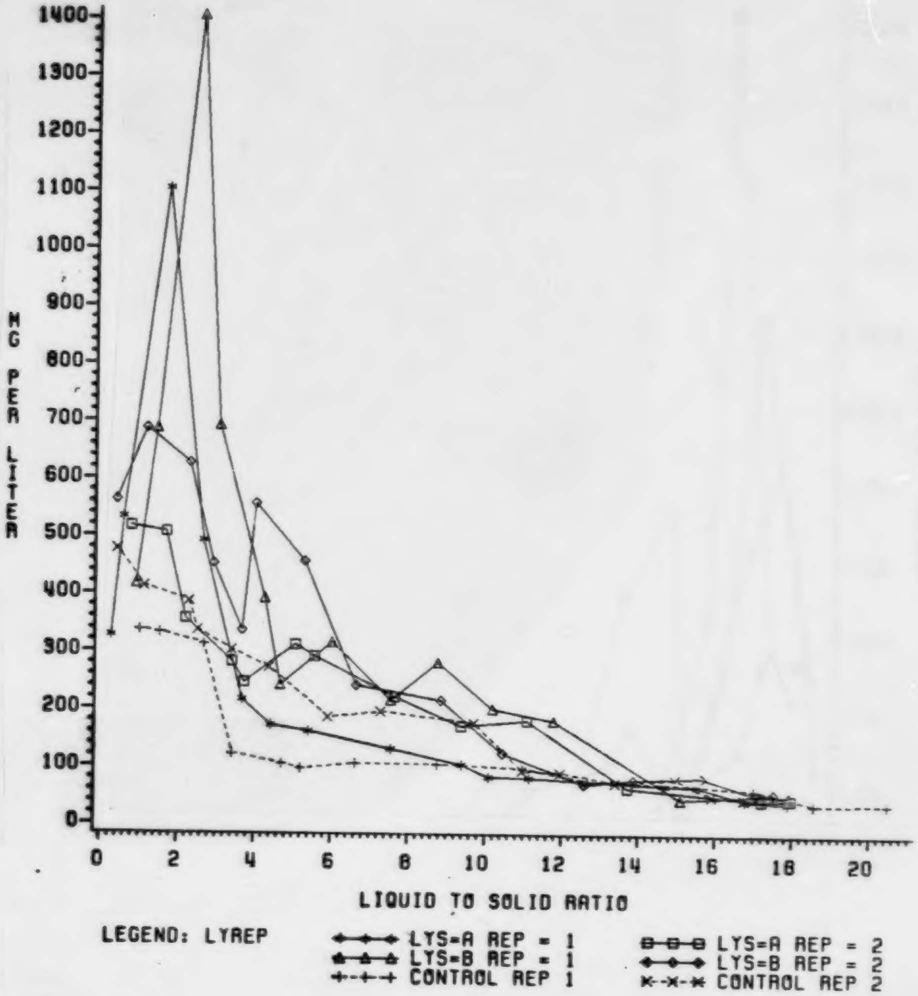

Fig. 23. Leaching of potassium from API/incinerator ash-Target.

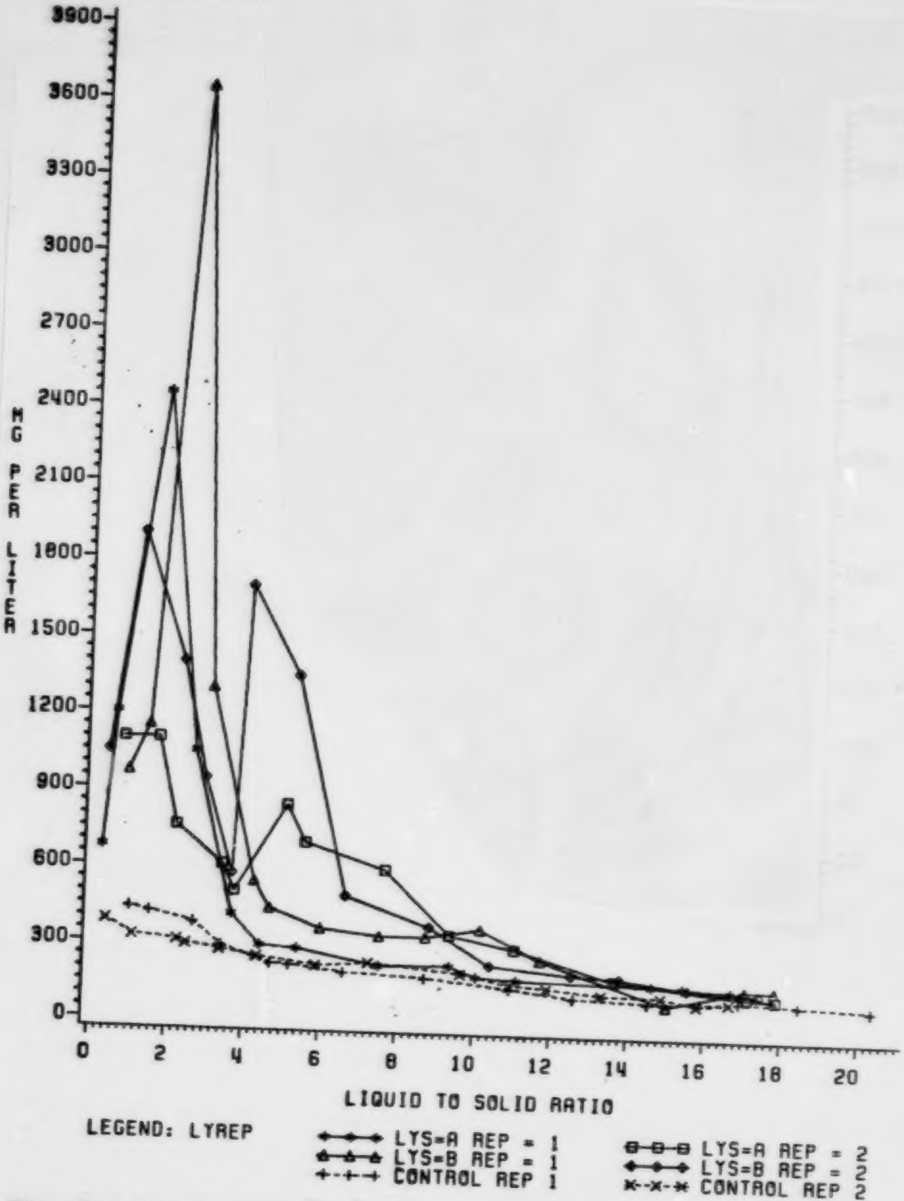

Fig. 24. Leaching of sodium from API/incinerator ash-Target.

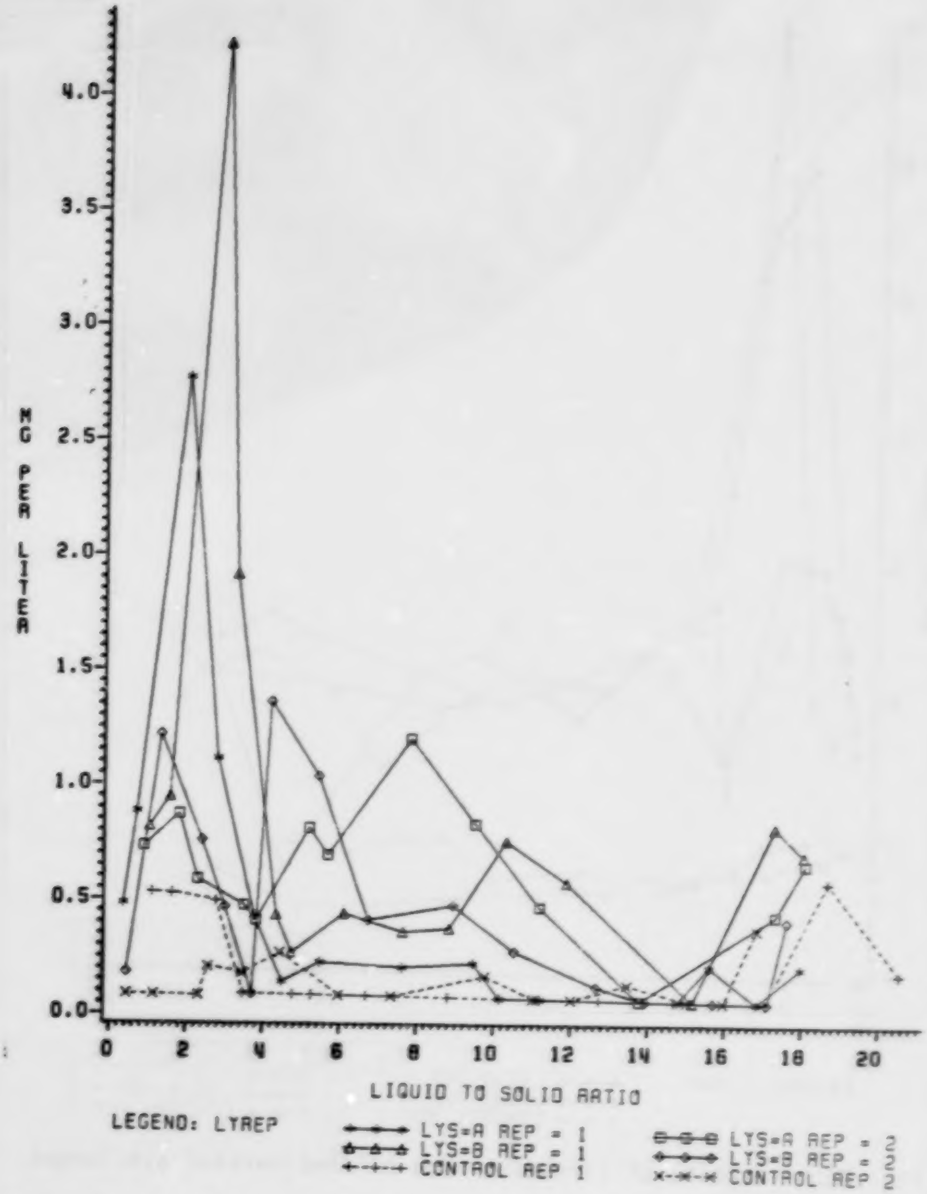

Fig. 25. Leaching of molybdenum from API/incinerator ash-Target.

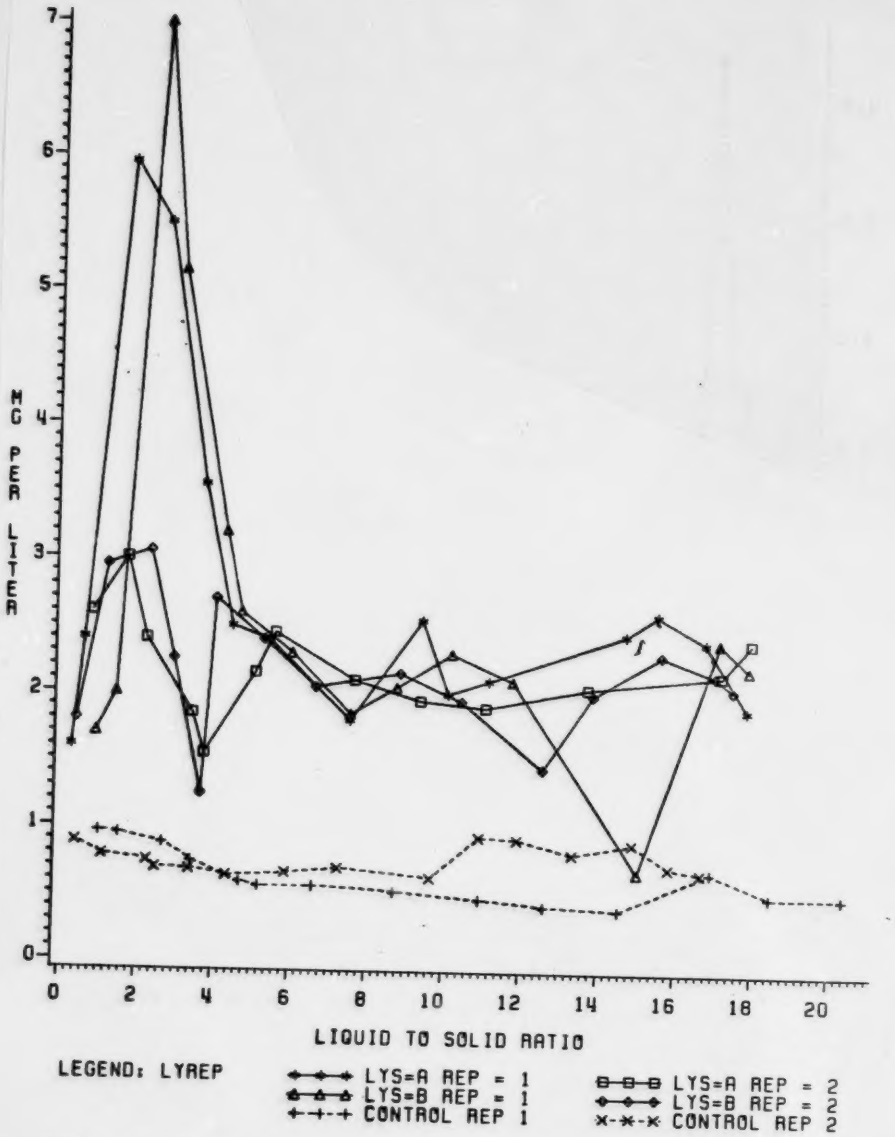

Fig. 26. Leaching of strontium from API/incinerator ash-Target.

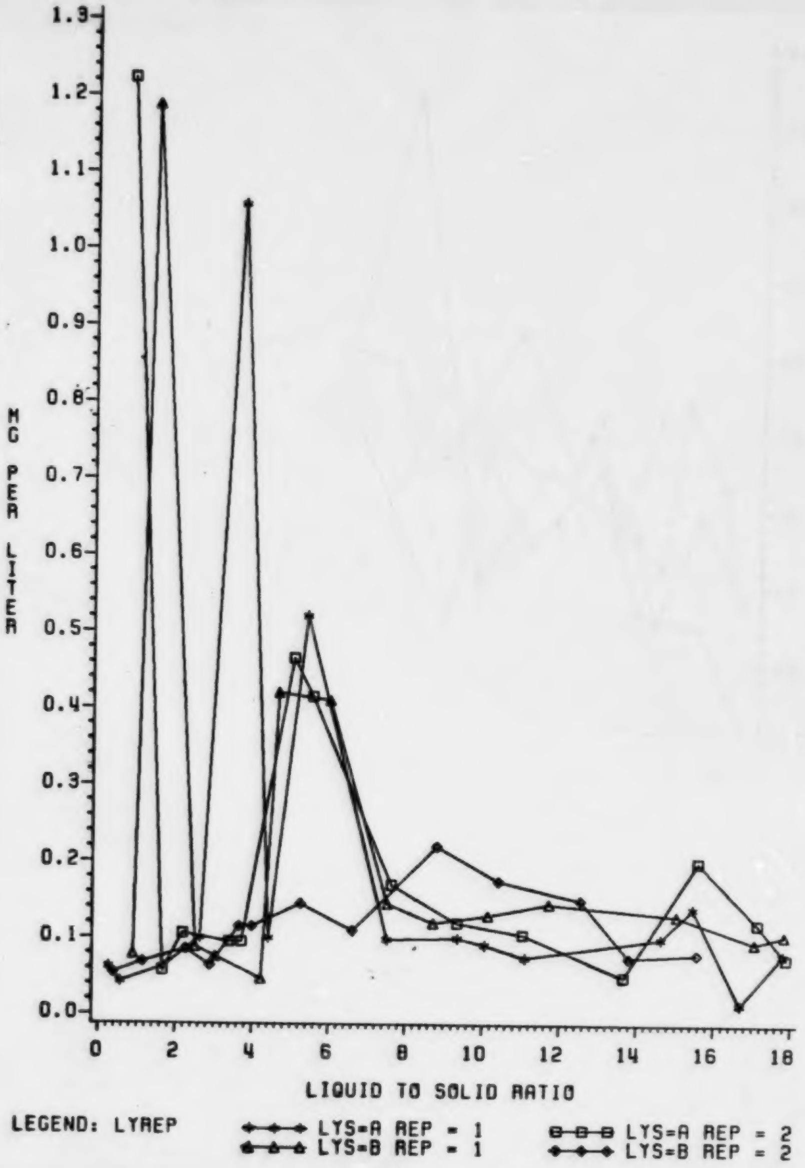

Fig. 27. Leaching of naphthalene from API/incinerator ash-Target.

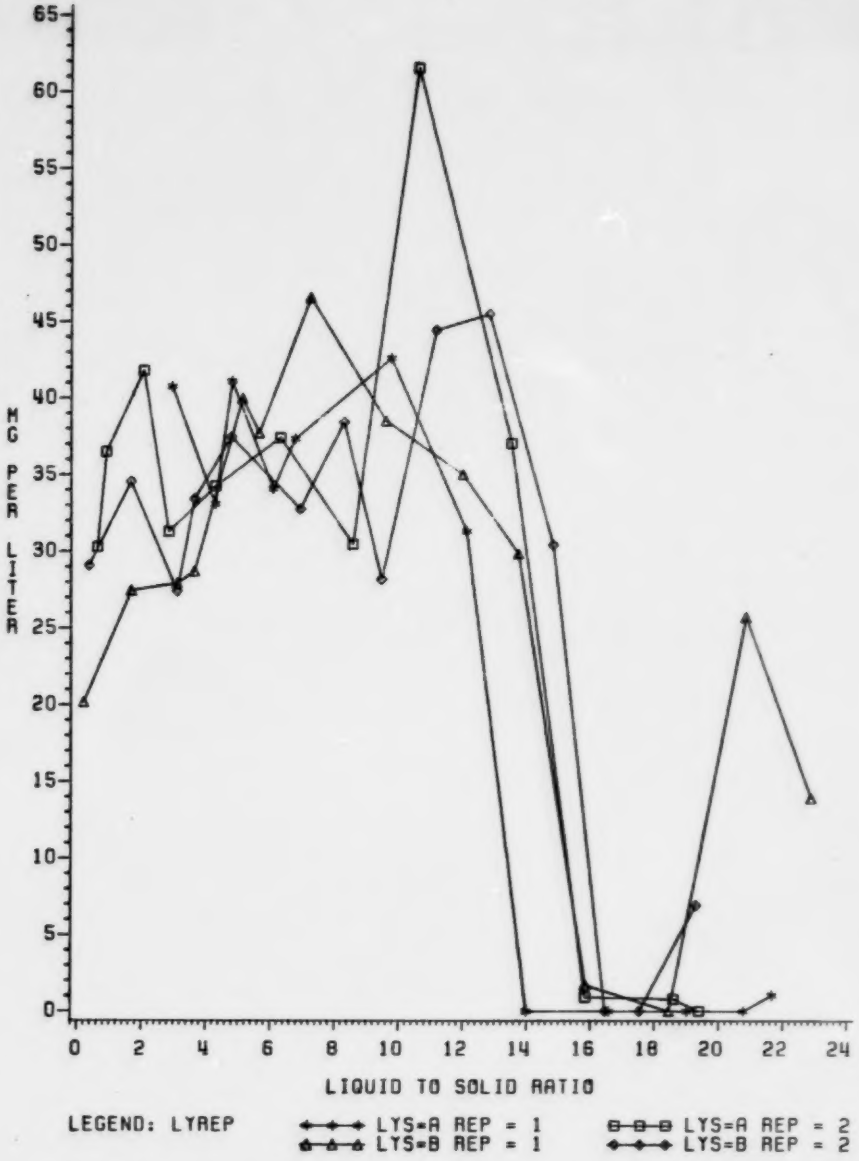

Fig. 28. Leaching of dichloroethane from dichloroethylene still bottoms-Target.

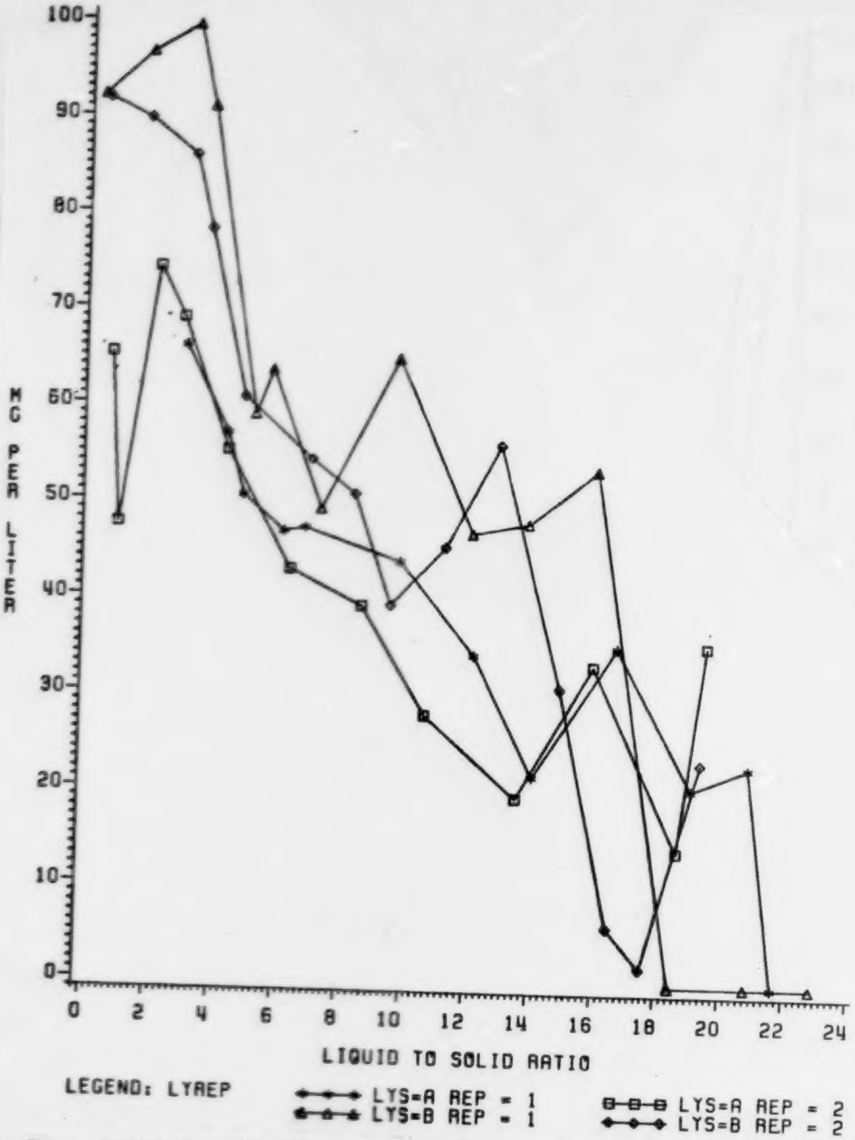

Fig. 29. Leaching of trichloroethane from dichloroethylene still bottoms-Target.

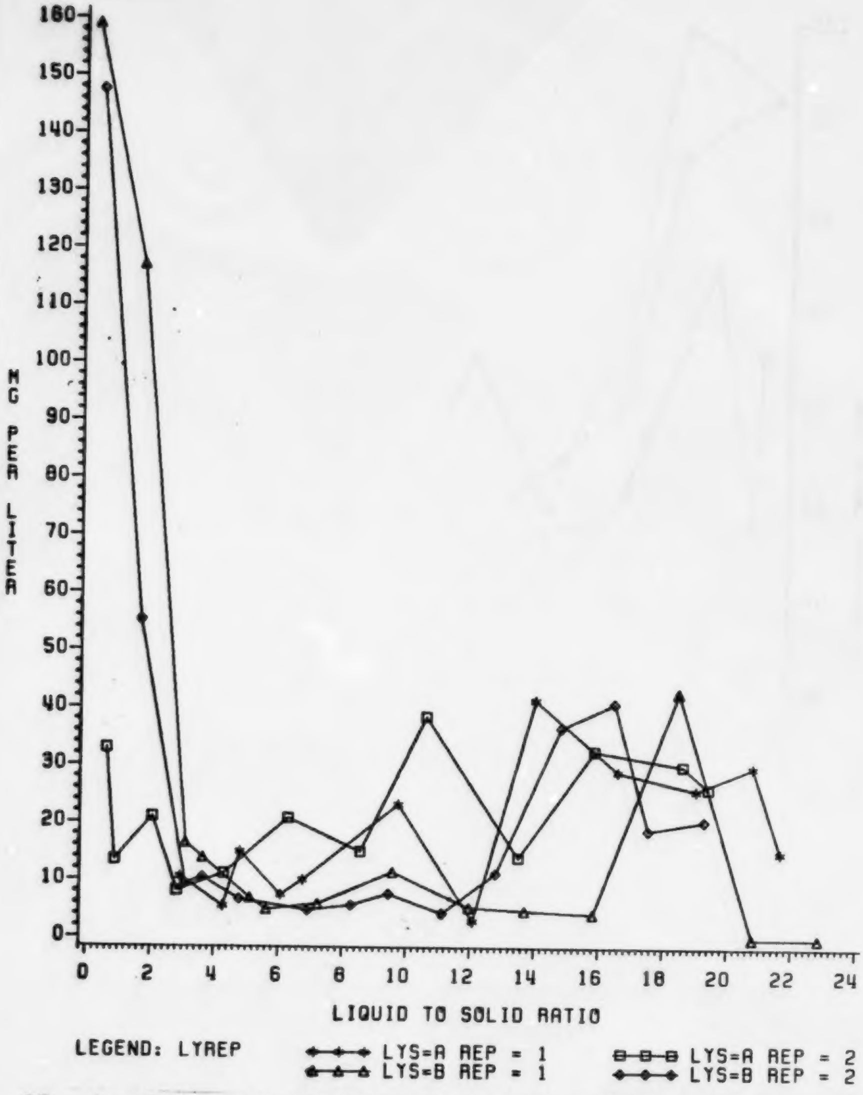

Fig. 30. **Leaching of trichloroethylene from dichloroethylene still bottoms-Target.**

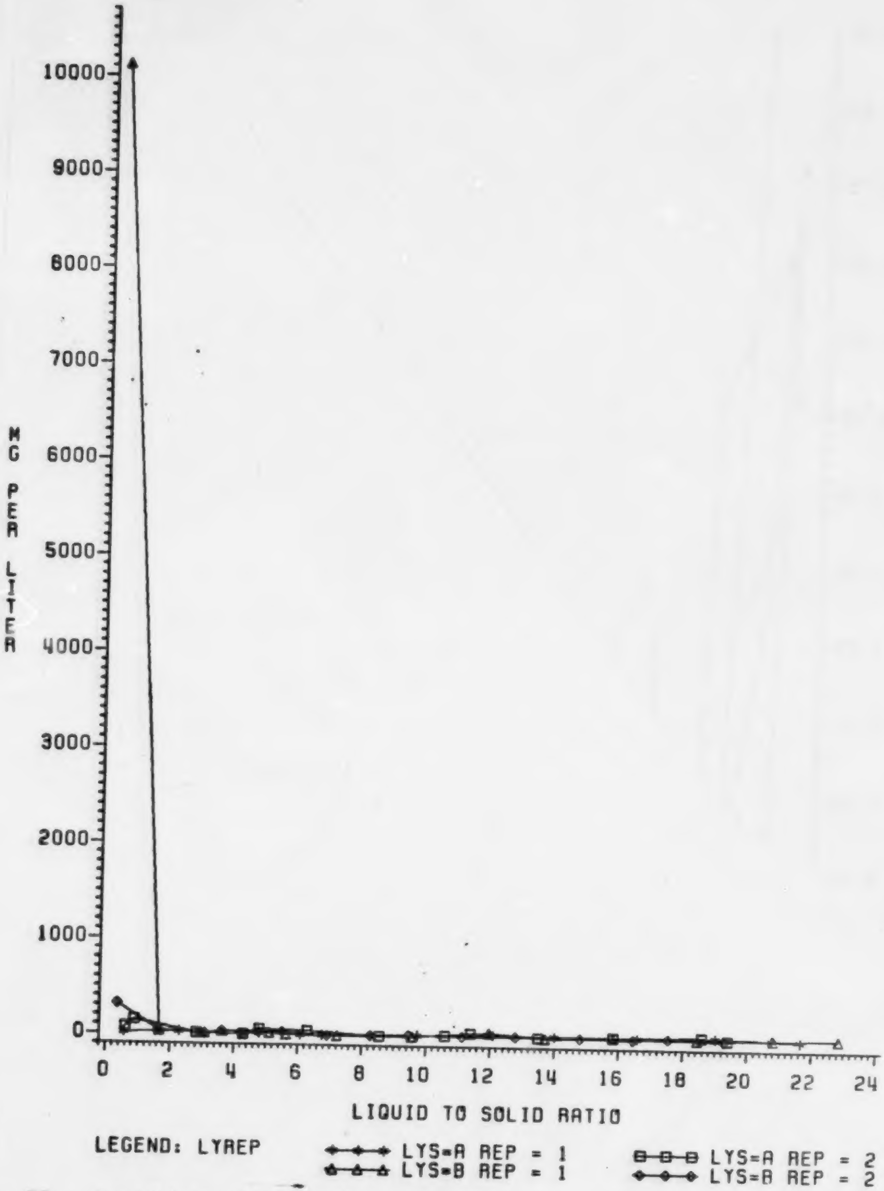

Fig. 31. Leaching of hexachlorobutadiene from dichloroethylene still bottoms-Target.

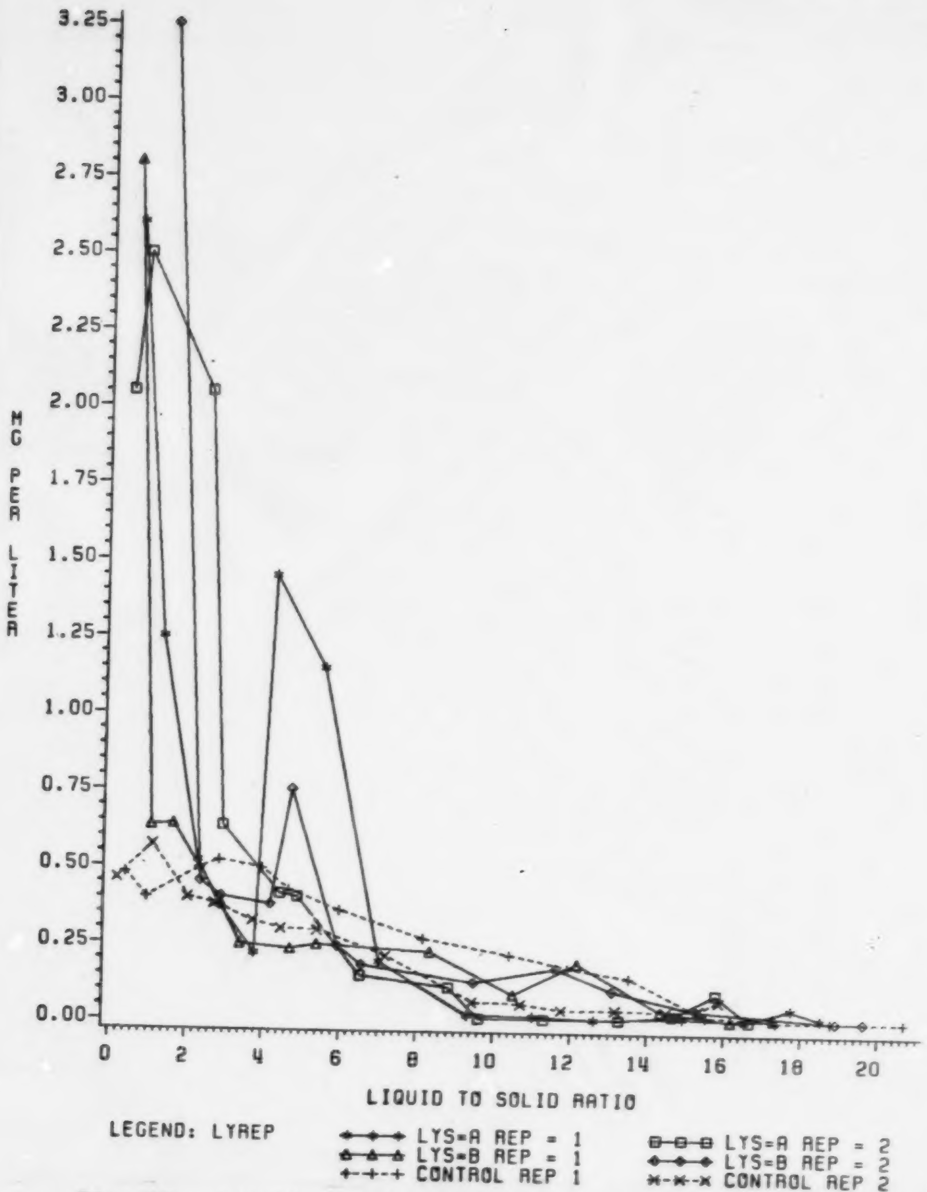

Fig. 32. Leaching of barium from paint sludge-Target.

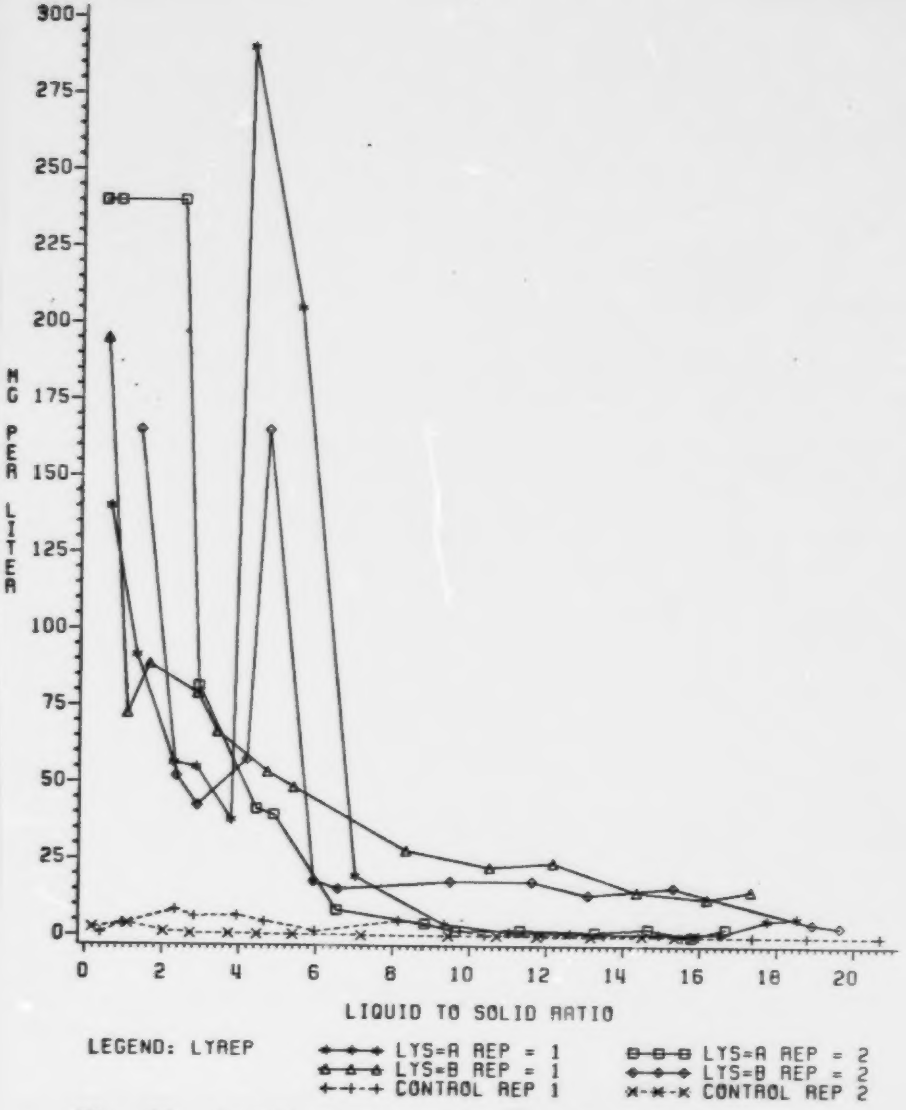

Fig. 33. Leaching of zinc from paint sludge-Target.

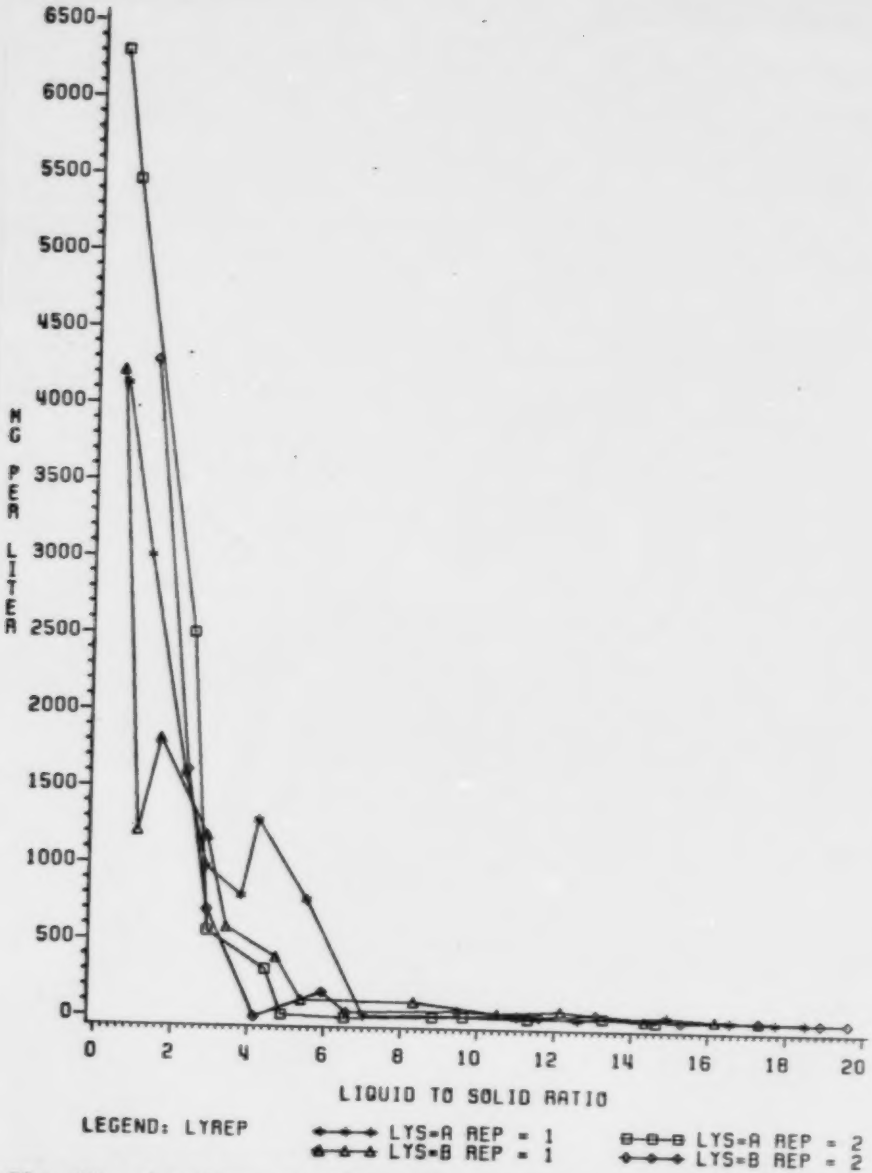

Fig. 34. Leaching of ethoxyethanol from paint sludge-Target.

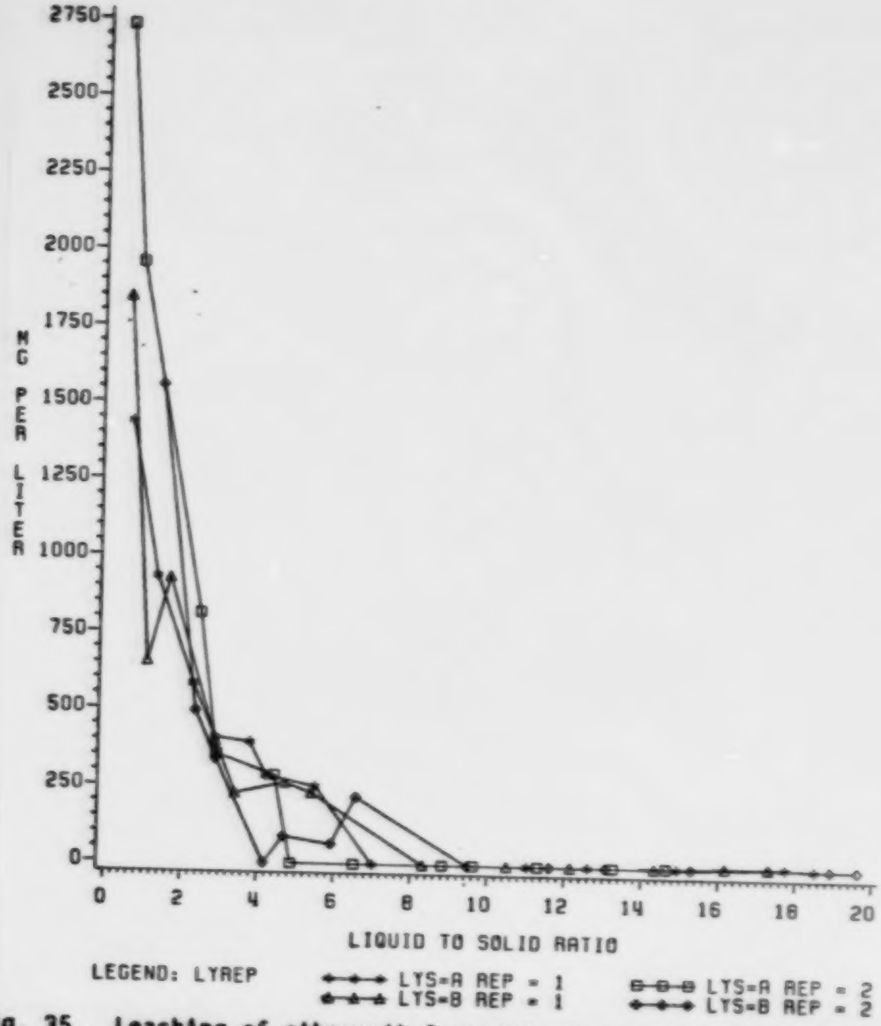

Fig. 35. Leaching of ethoxyethyl acetate from paint sludge-Target.

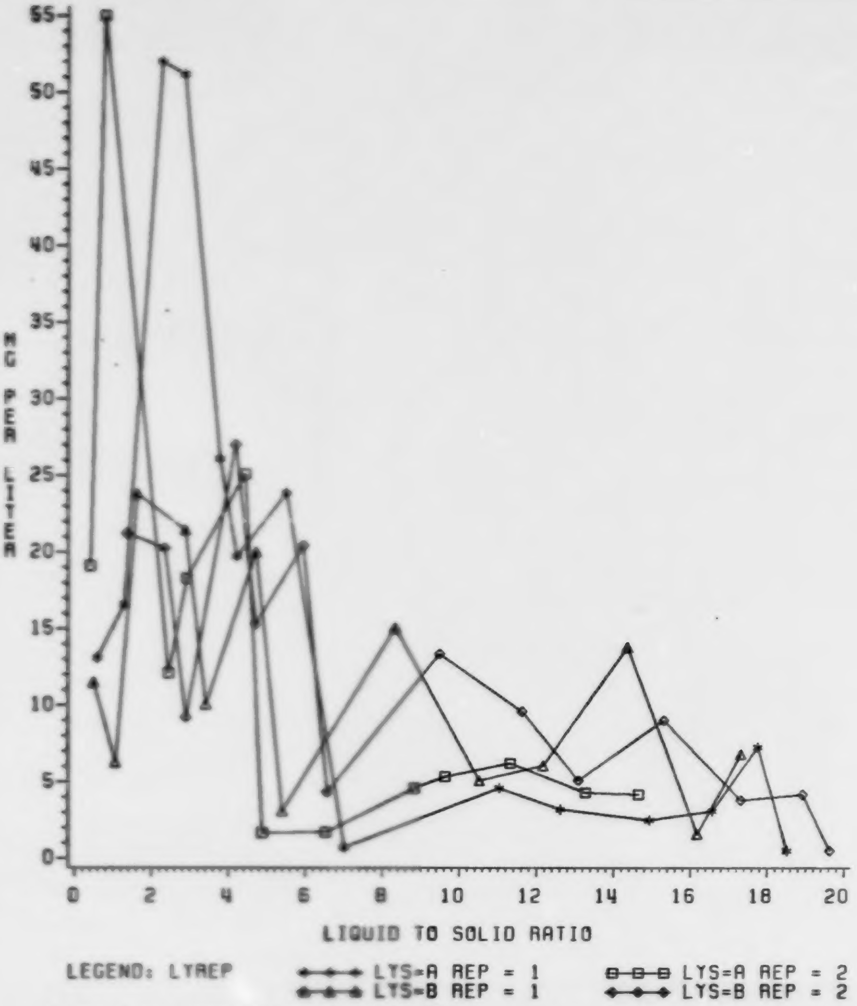

Fig. 36. Leaching of toluene from paint sludge-Target.

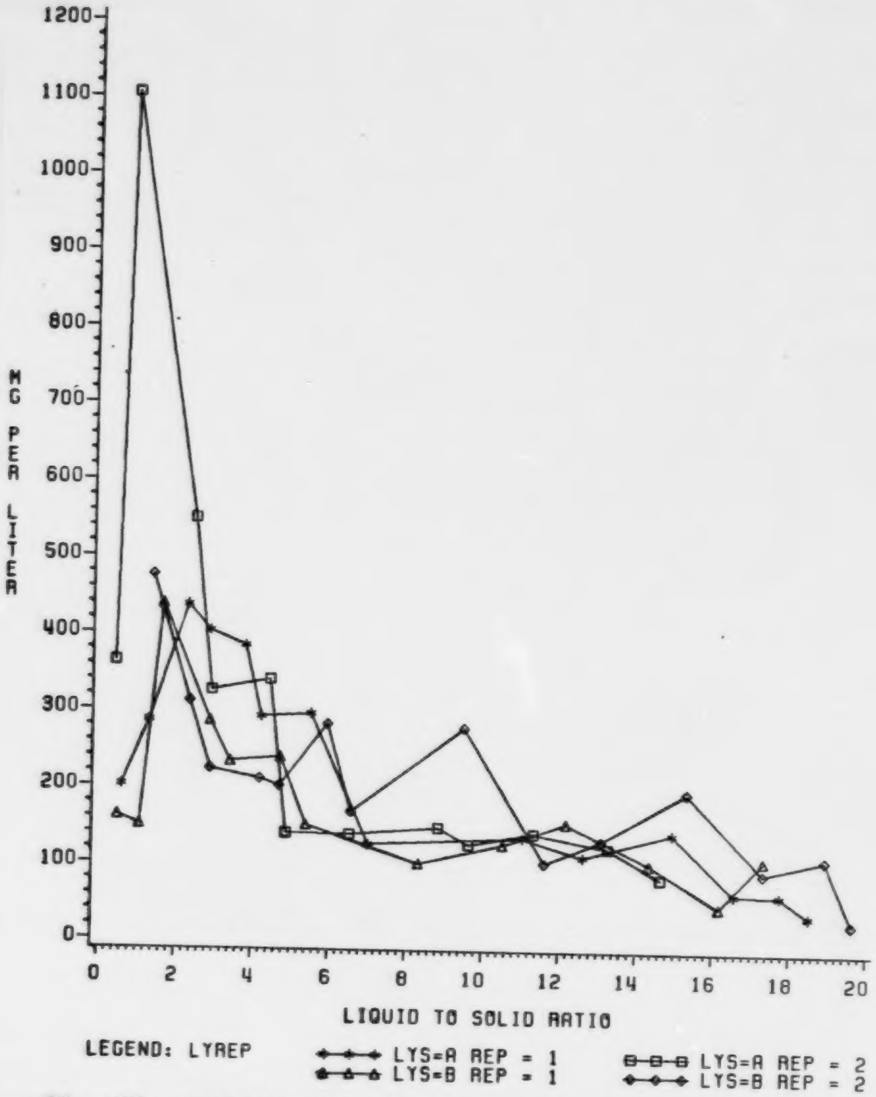

Fig. 37. Leaching of xylenes from paint sludge-Target.

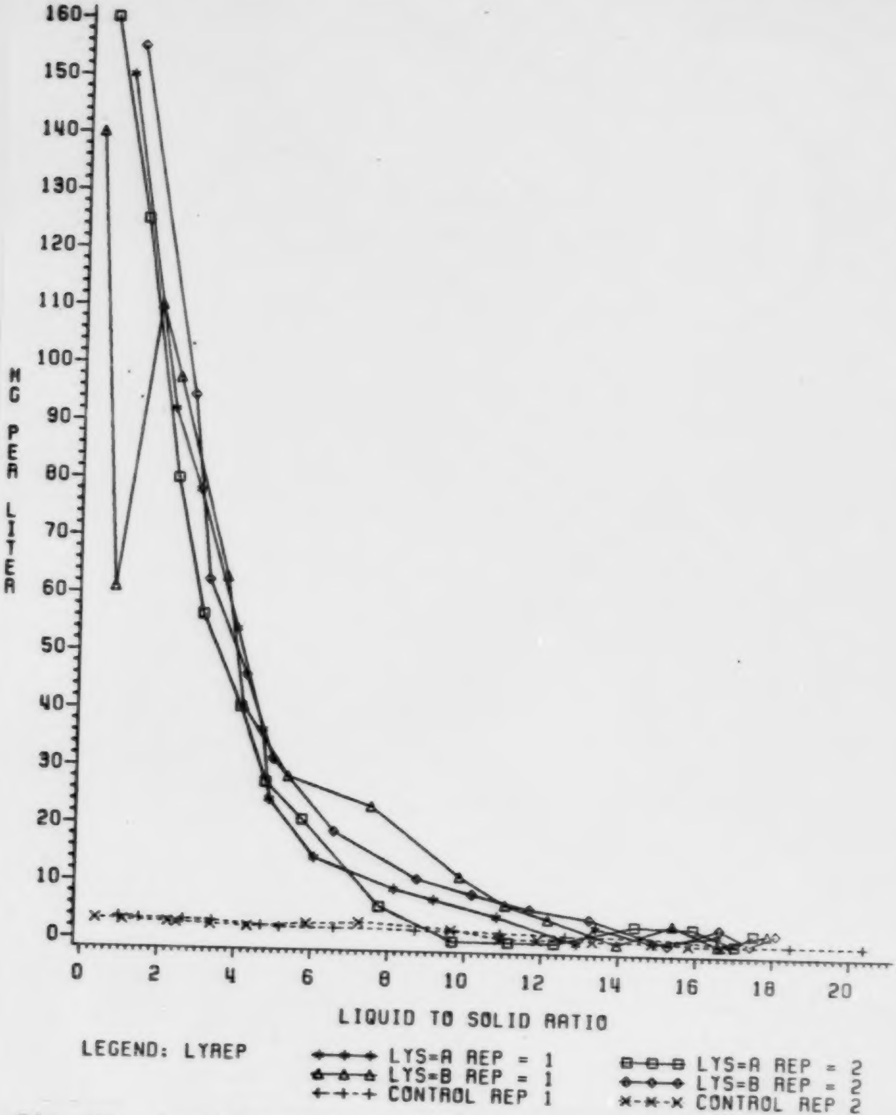

Fig. 38. Leaching of boron from electroplating waste-Target.

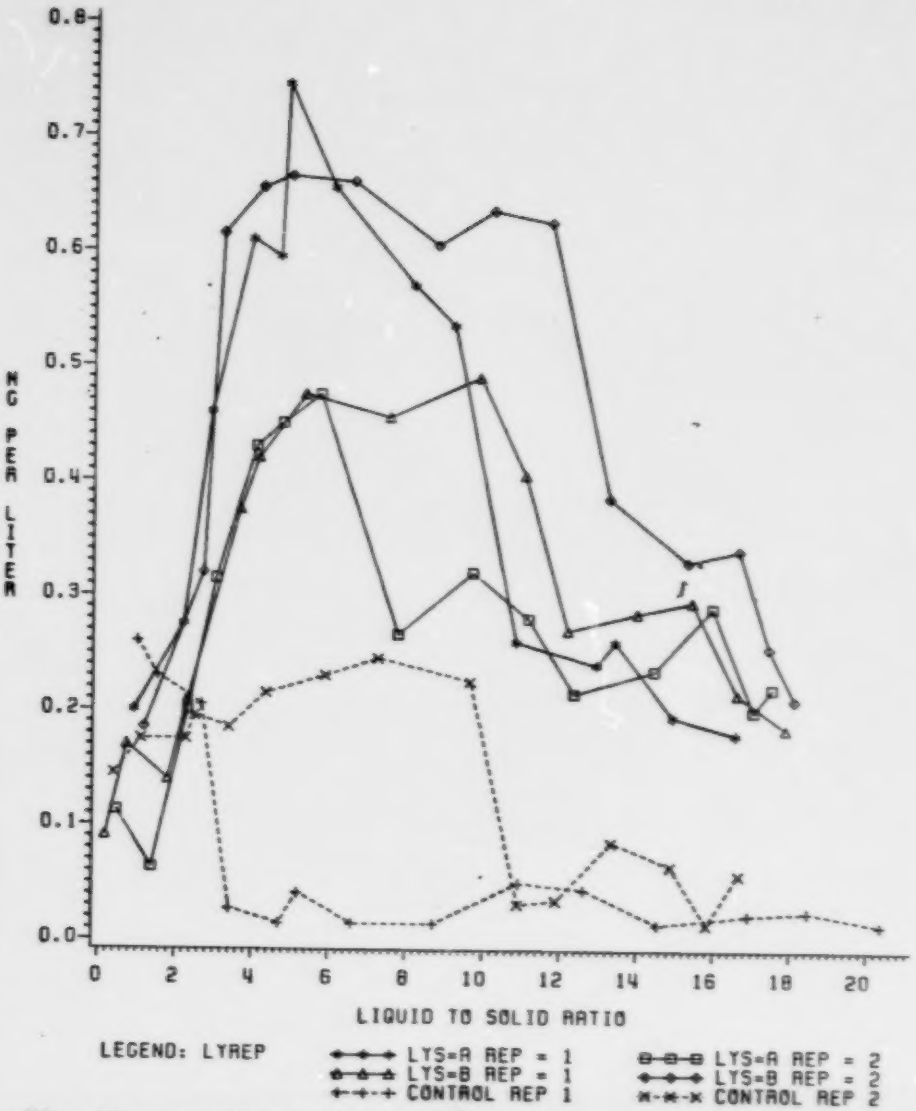

Fig. 39. Leaching of barium from electroplating waste-Target.

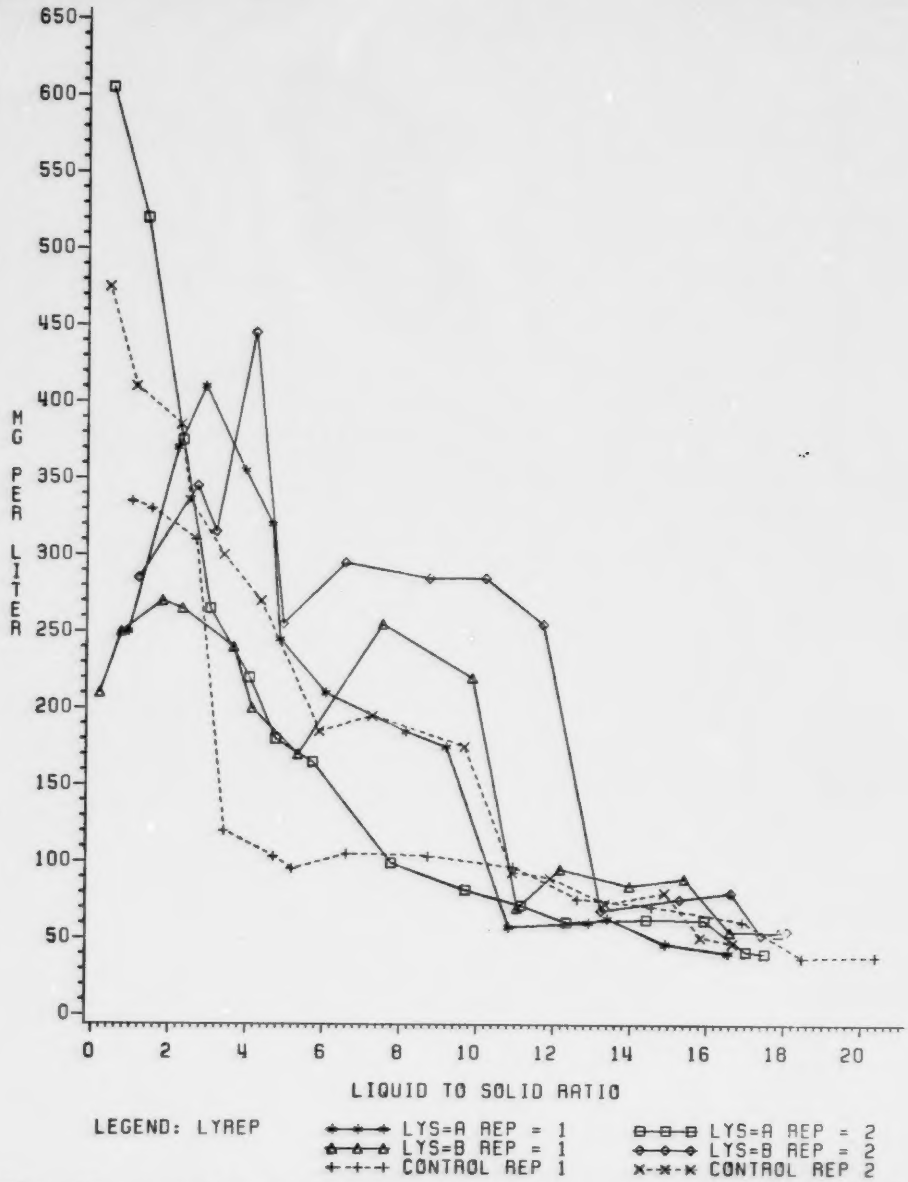

Fig. 40. Leaching of potassium from electroplating waste-Target.

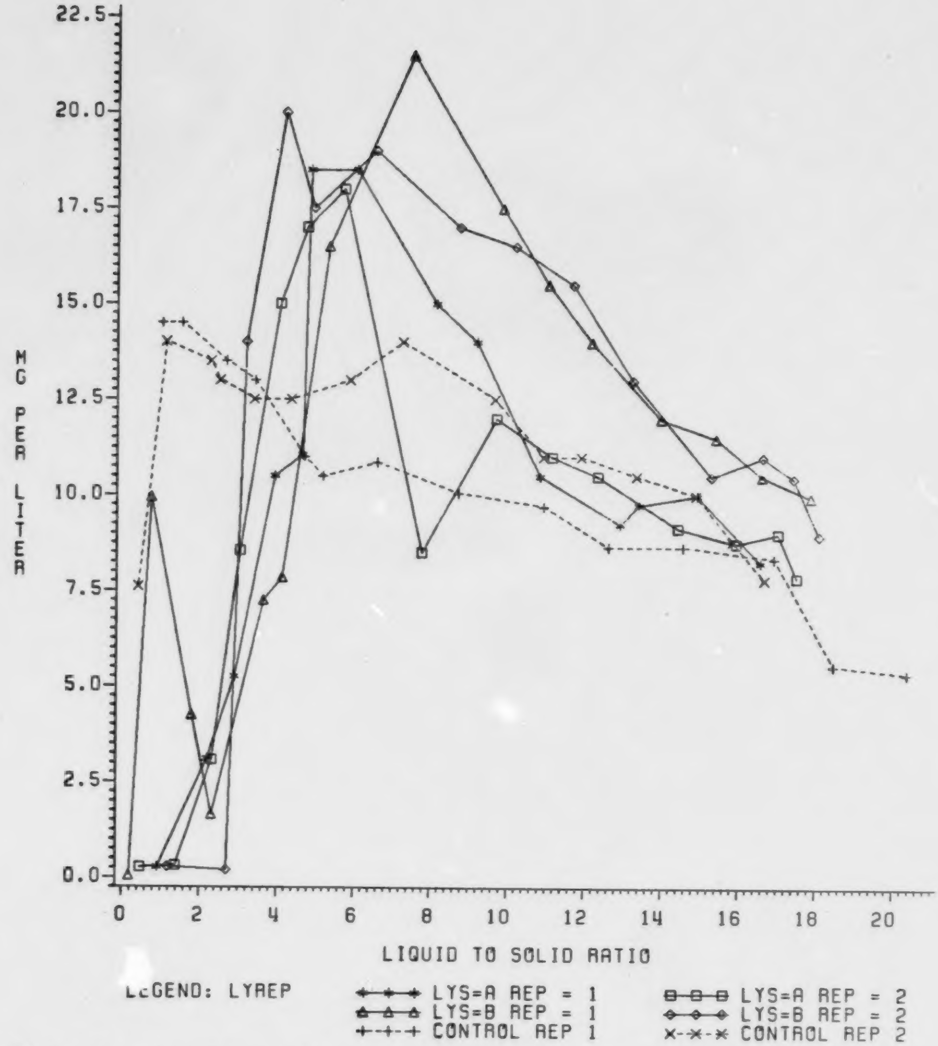

Fig. 41. Leaching of manganese from electroplating waste-Target.

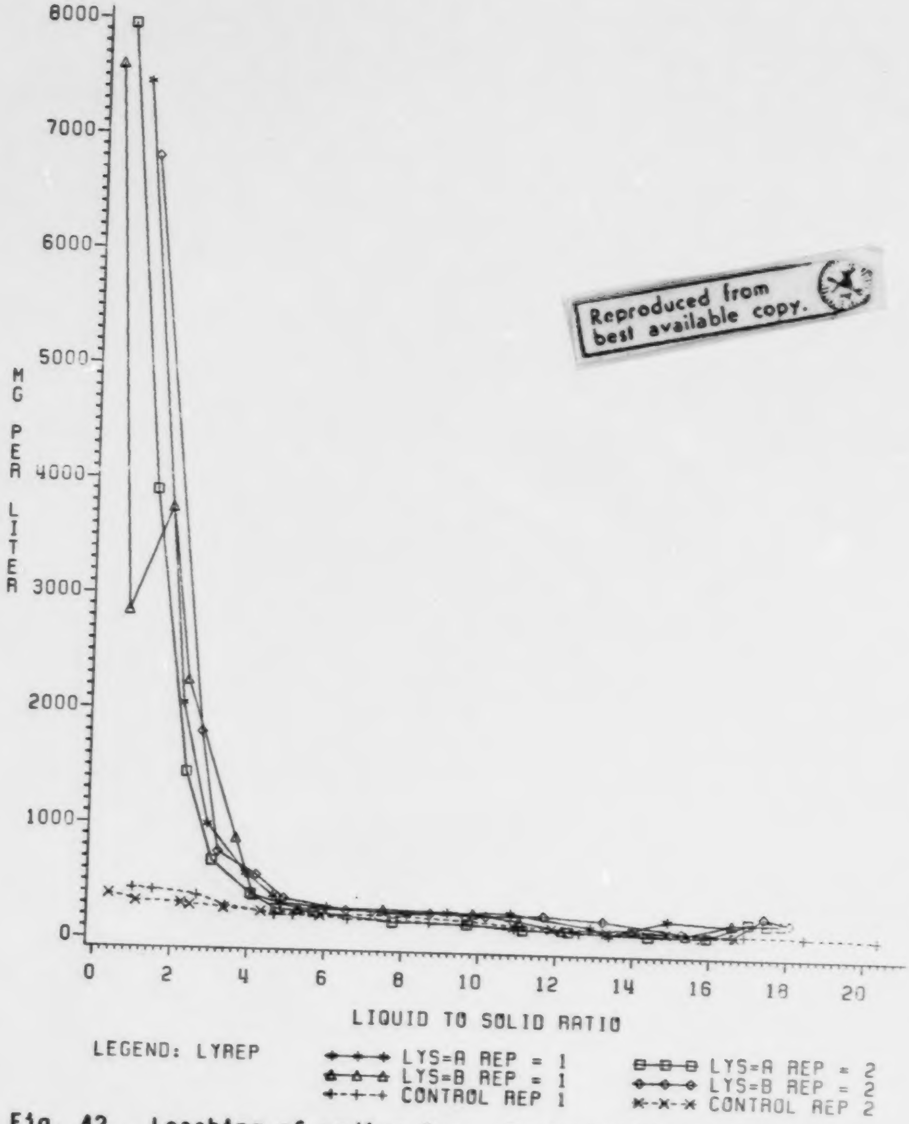

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Fig. 42. Leaching of sodium from electroplating waste-Target.

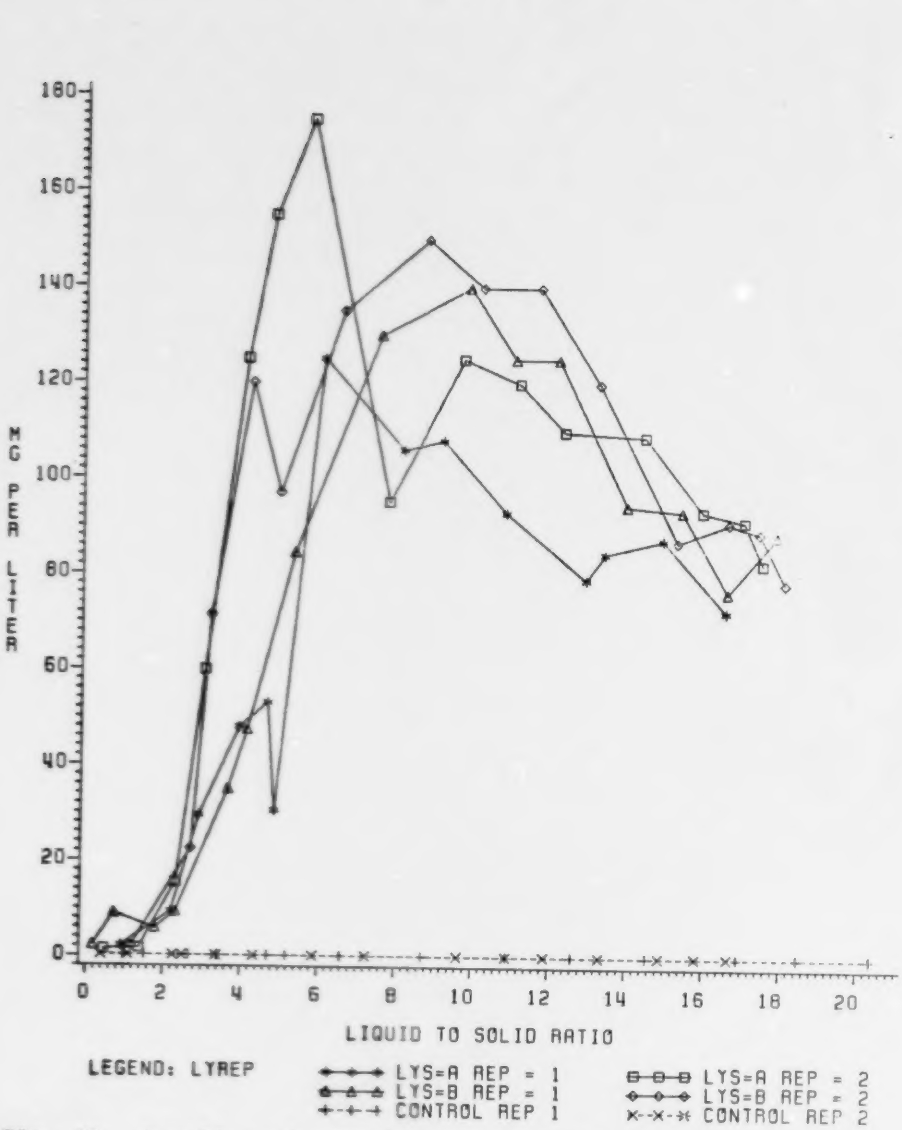

Fig. 43. Leaching of nickel from electroplating waste-Target.

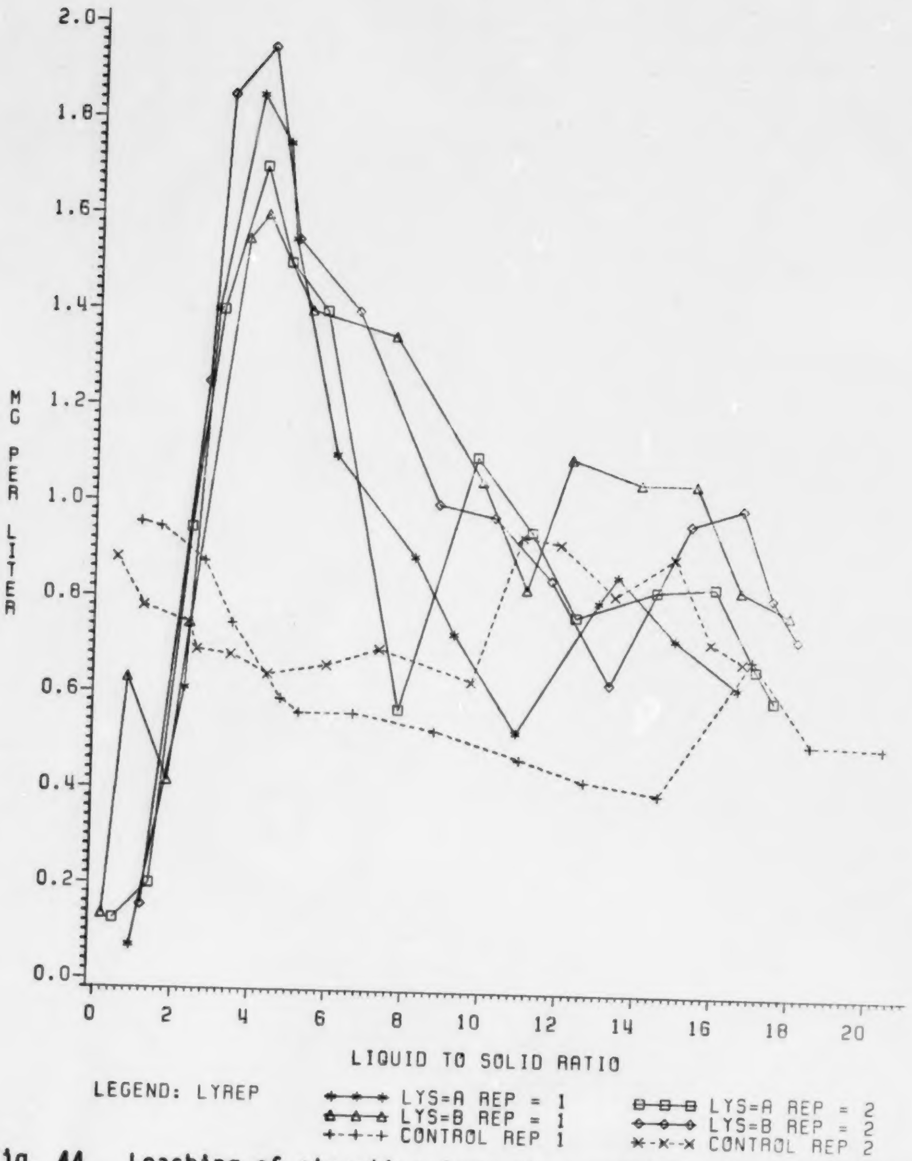

Fig. 44. Leaching of strontium from electroplating waste-Target.

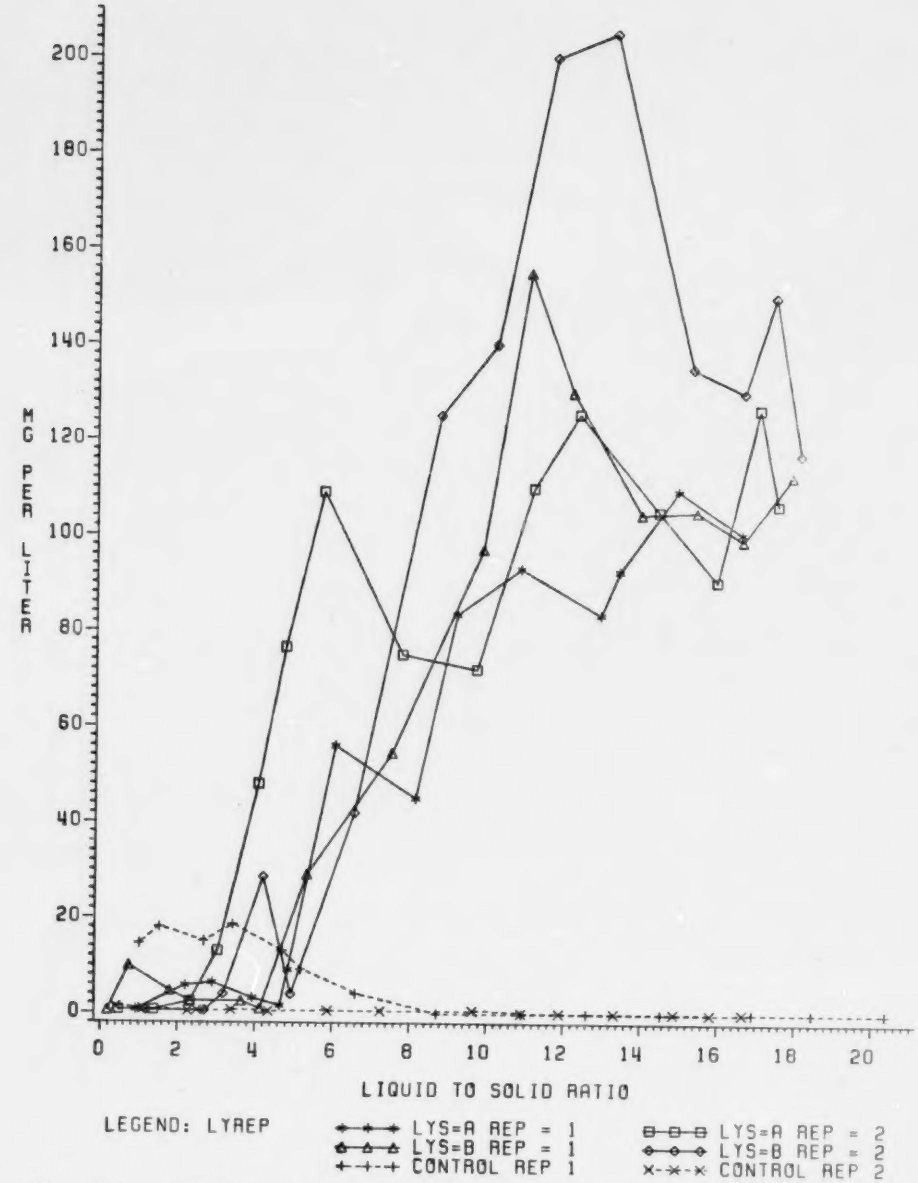

Fig. 45. Leaching of zinc from electroplating waste-Target.

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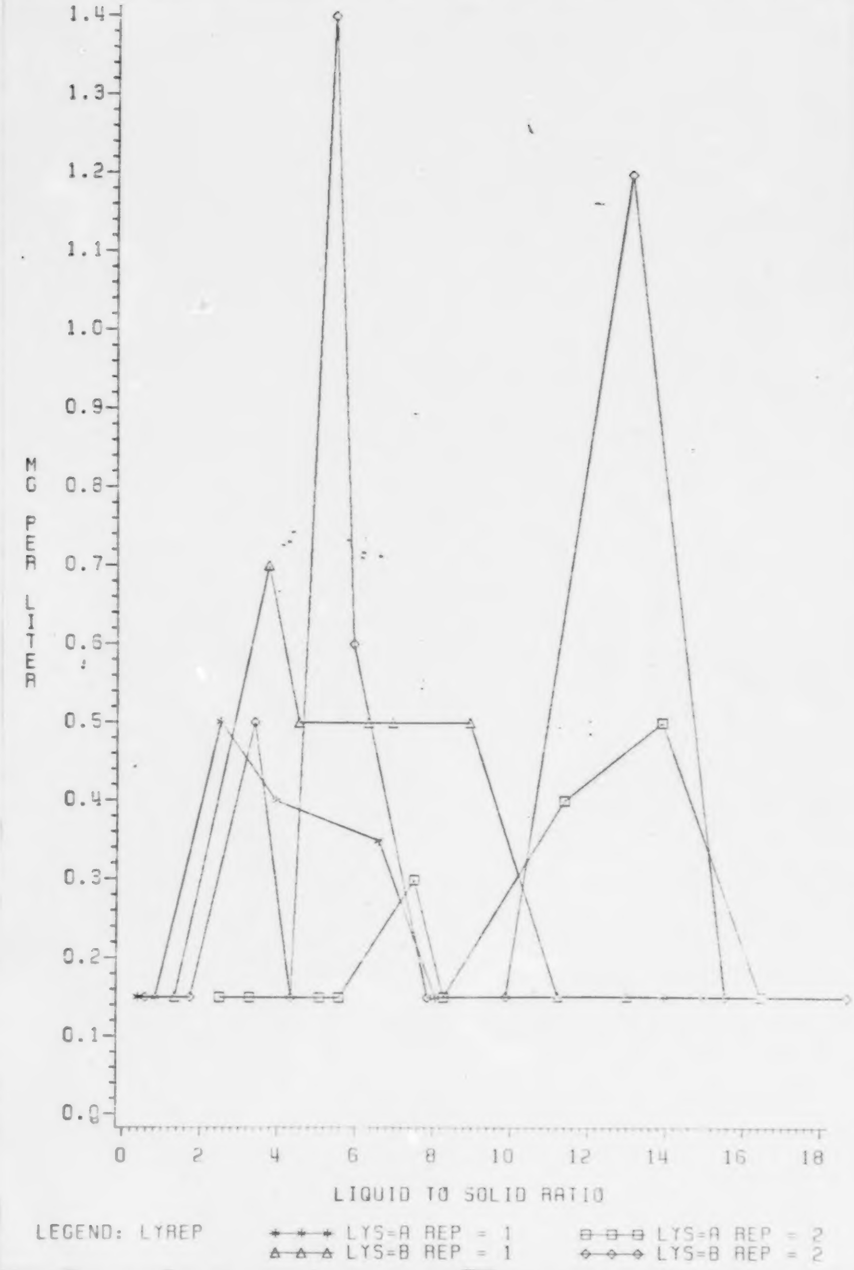

Fig. 46. Leaching of toluene from API/incinerator ash-Target.

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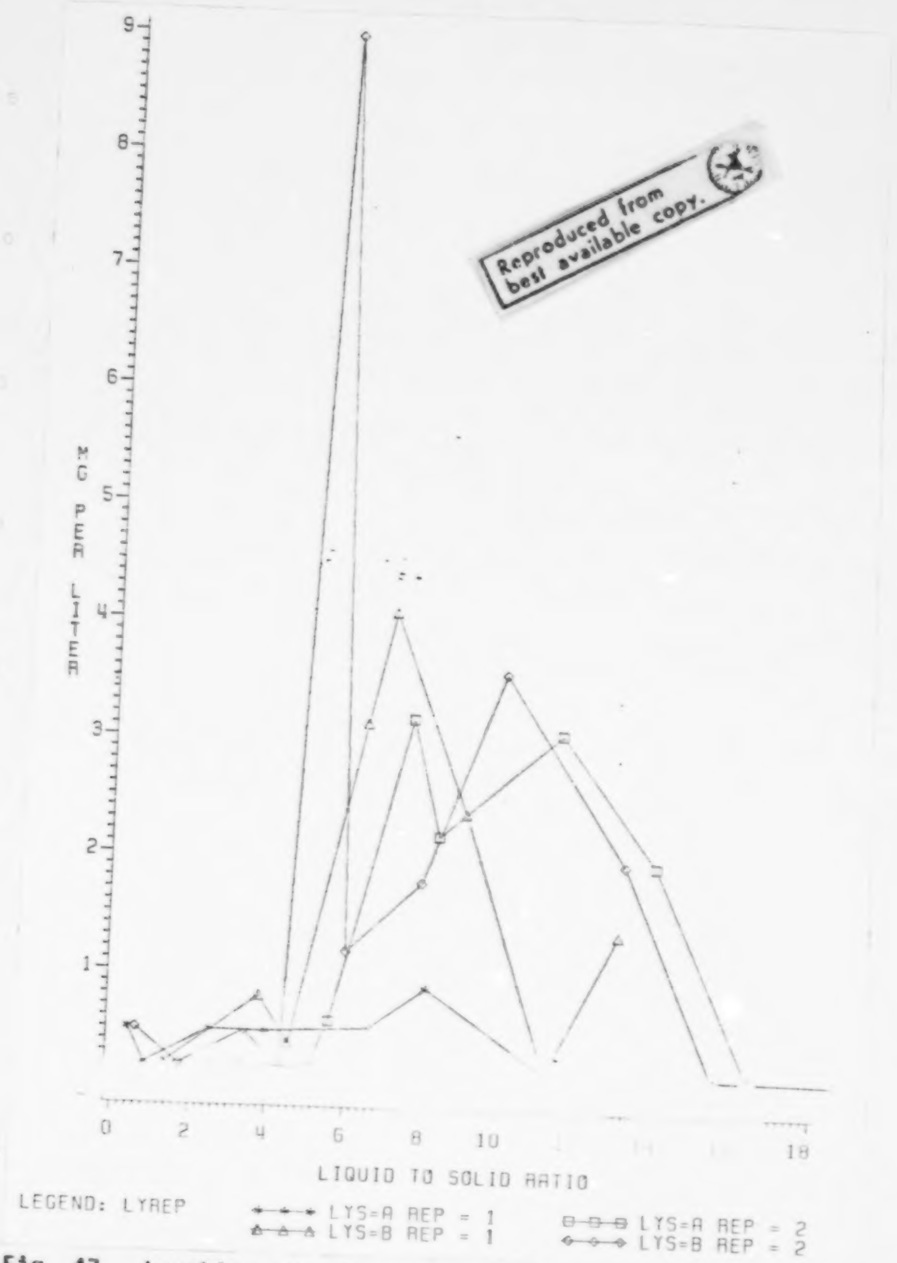

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Fig. 47. Leaching of xylenes from API/incinerator ash-Target.

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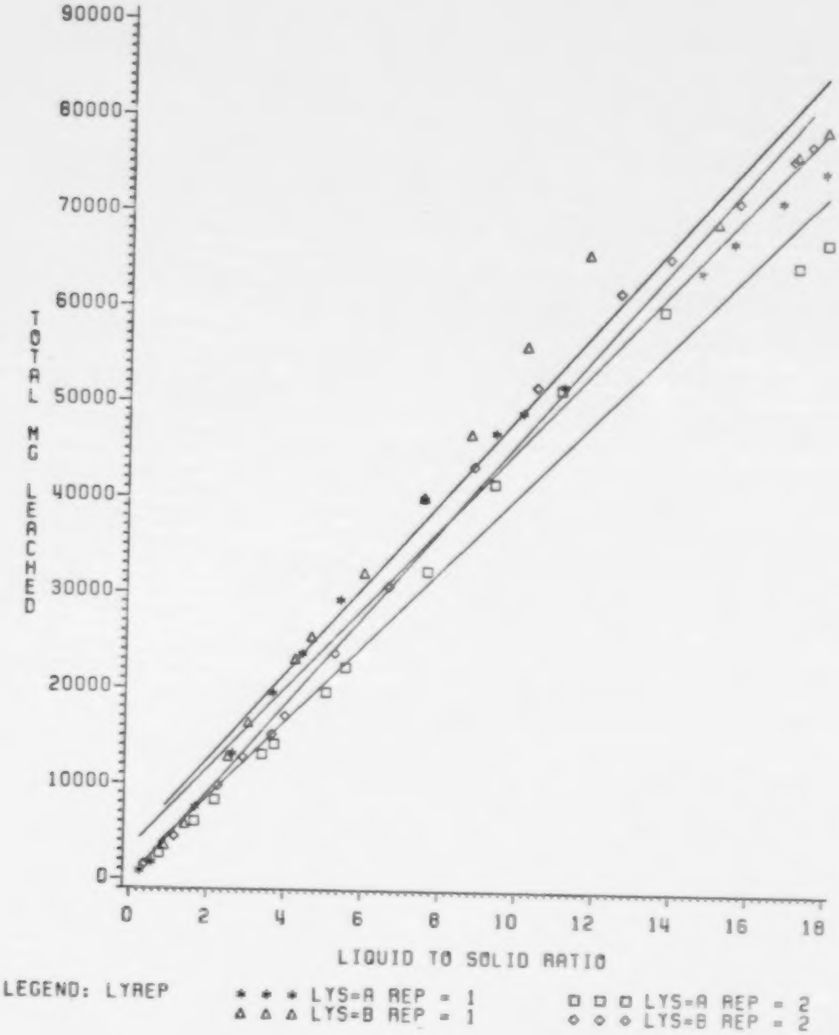

Fig. 48. Fitted accumulative leaching curves of calcium from API/incinerator ash in Target, expressed in terms of liquid/solid ratios.

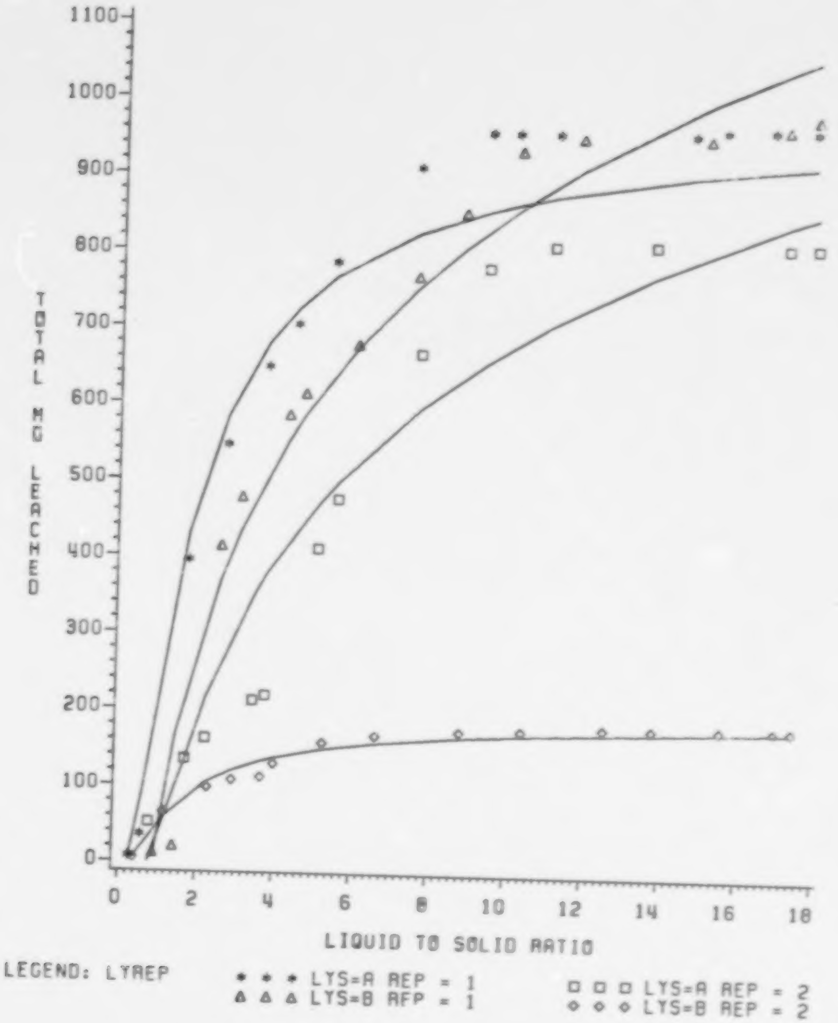

Fig. 49. Fitted accumulative leaching curves of chromium from API/incinerator ash in Target, expressed in terms of liquid/solid ratios.

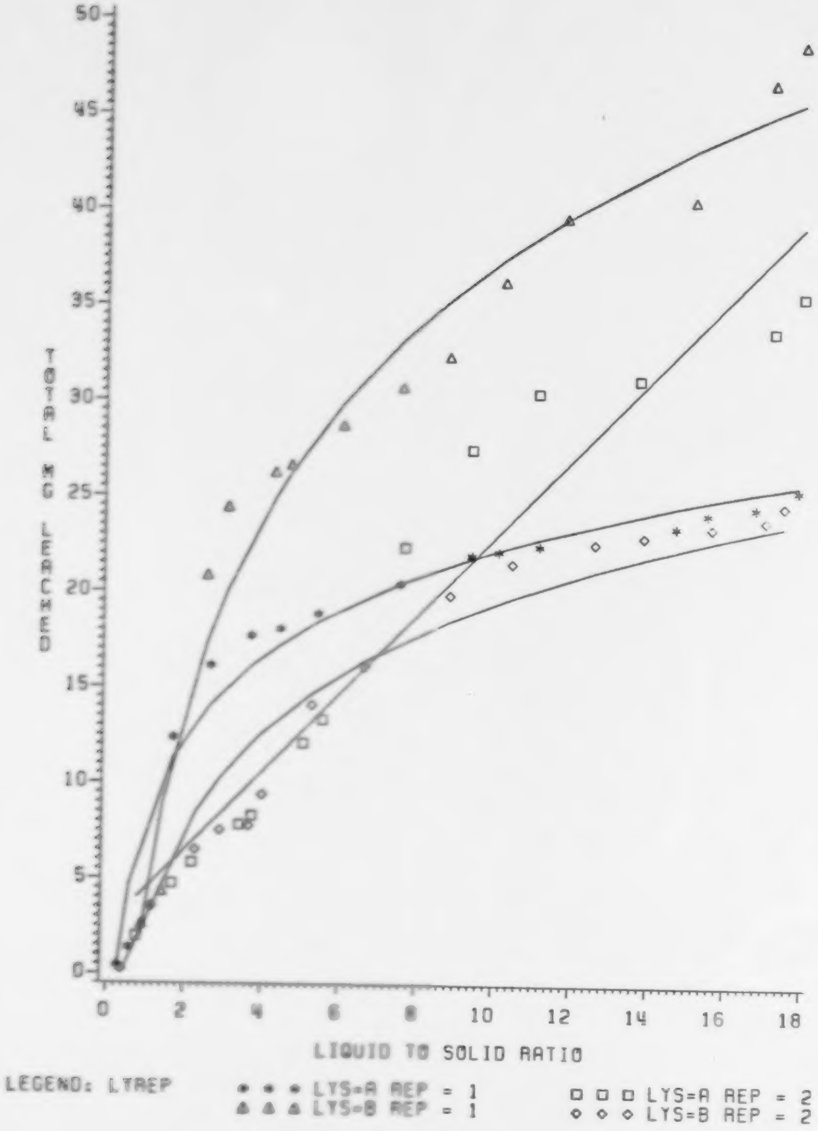

Fig. 50. Fitted accumulative leaching curves of molybdenum from API/incinerator ash in Target, expressed in terms of liquid/solid ratios.

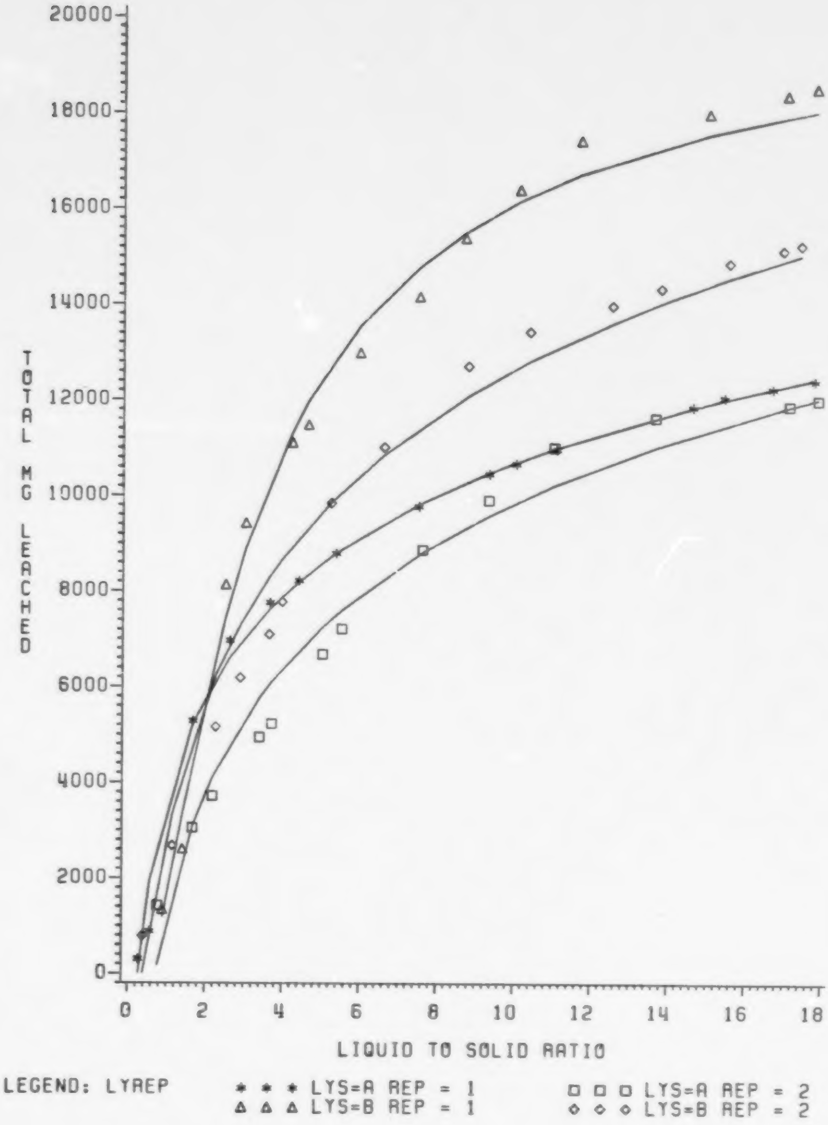

Fig. 51. Fitted accumulative leaching curves of potassium from API/incinerator ash in Target, expressed in terms of liquid/solid ratios.

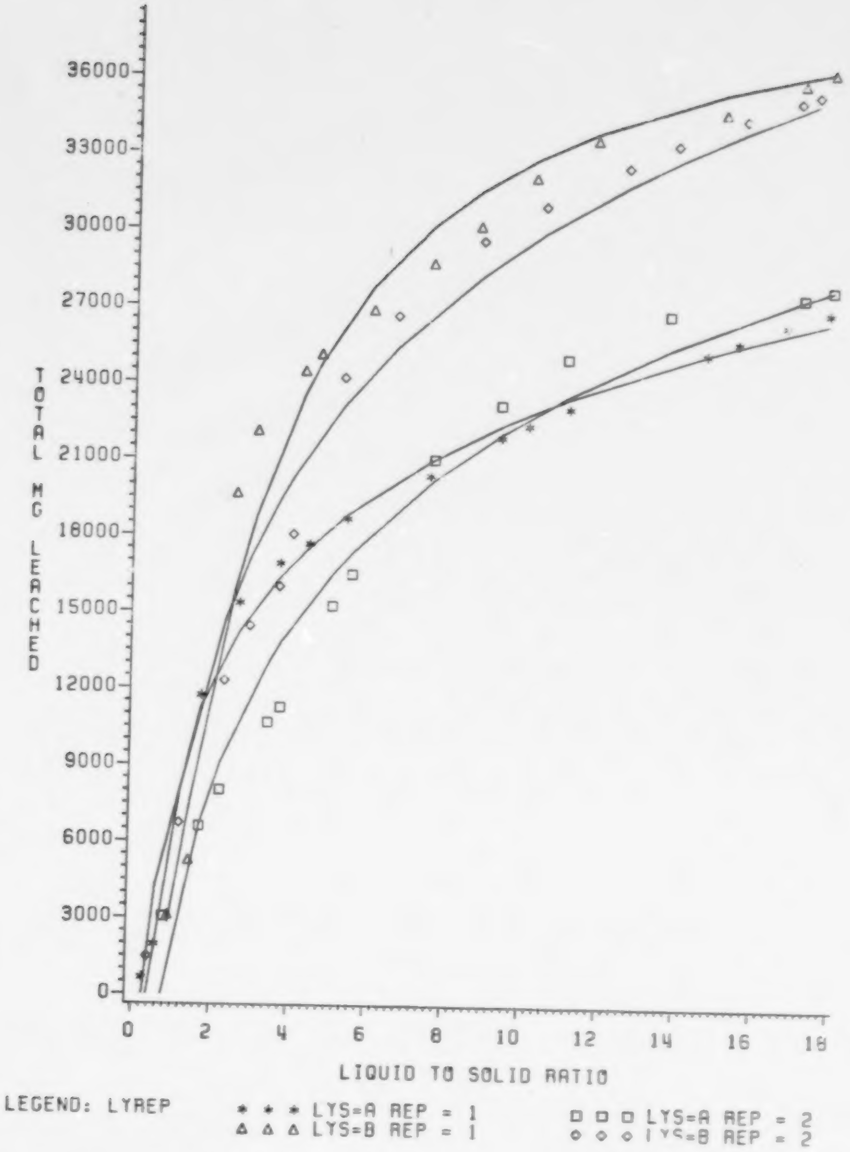

Fig. 52. Fitted accumulative leaching curves of sodium from API/incinerator ash in Target, expressed in terms of liquid/solid ratios.

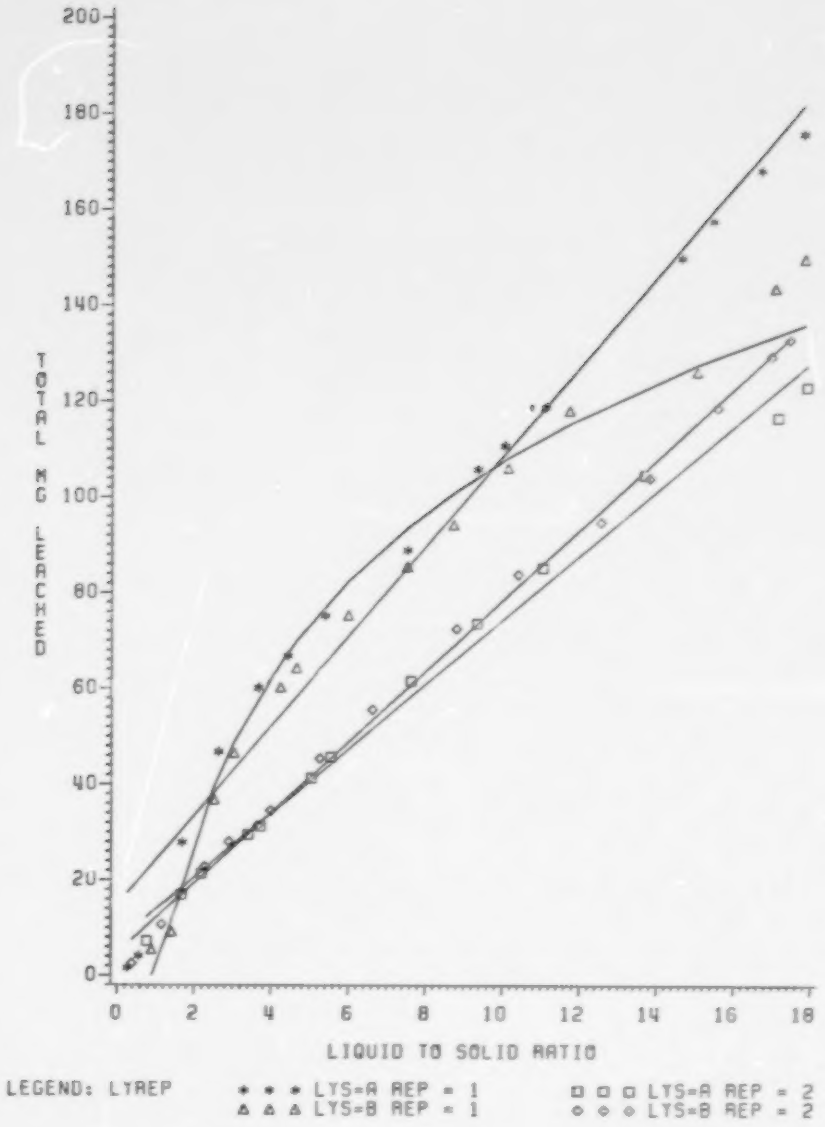

Fig. 53. Fitted accumulative leaching curves of strontium from API/incinerator ash in Target, expressed in terms of liquid/solid ratios.

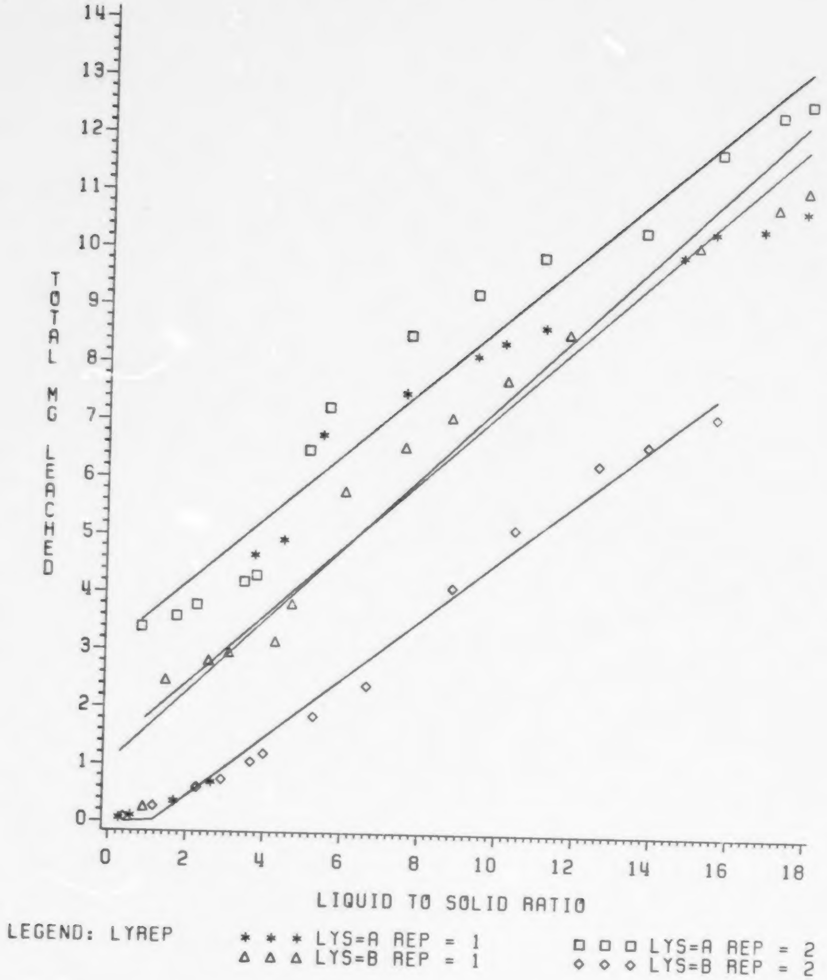

Fig. 54. Fitted accumulative leaching curves of naphthalene from API/incinerator ash in Target, expressed in terms of liquid/solid ratios.

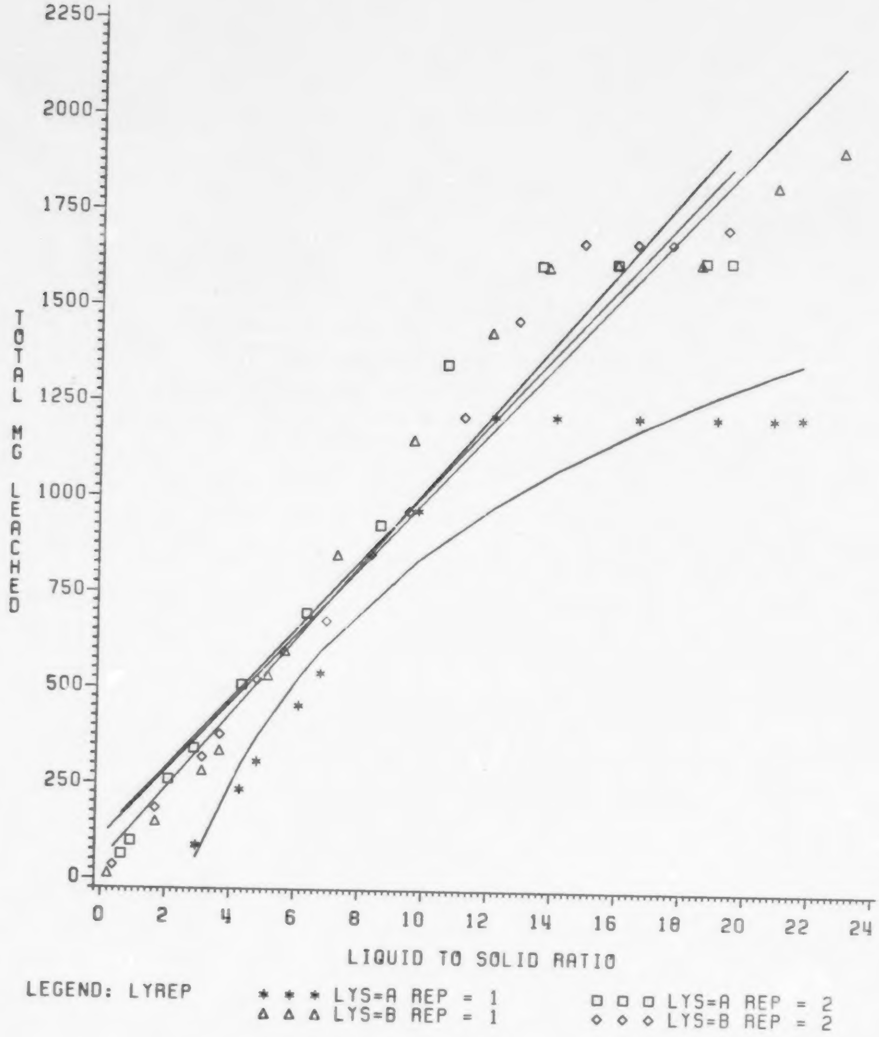

Fig. 55. Fitted accumulative leaching curves of dichloroethane from dichloroethylene still bottoms in Target, expressed in terms of liquid/solid ratios.

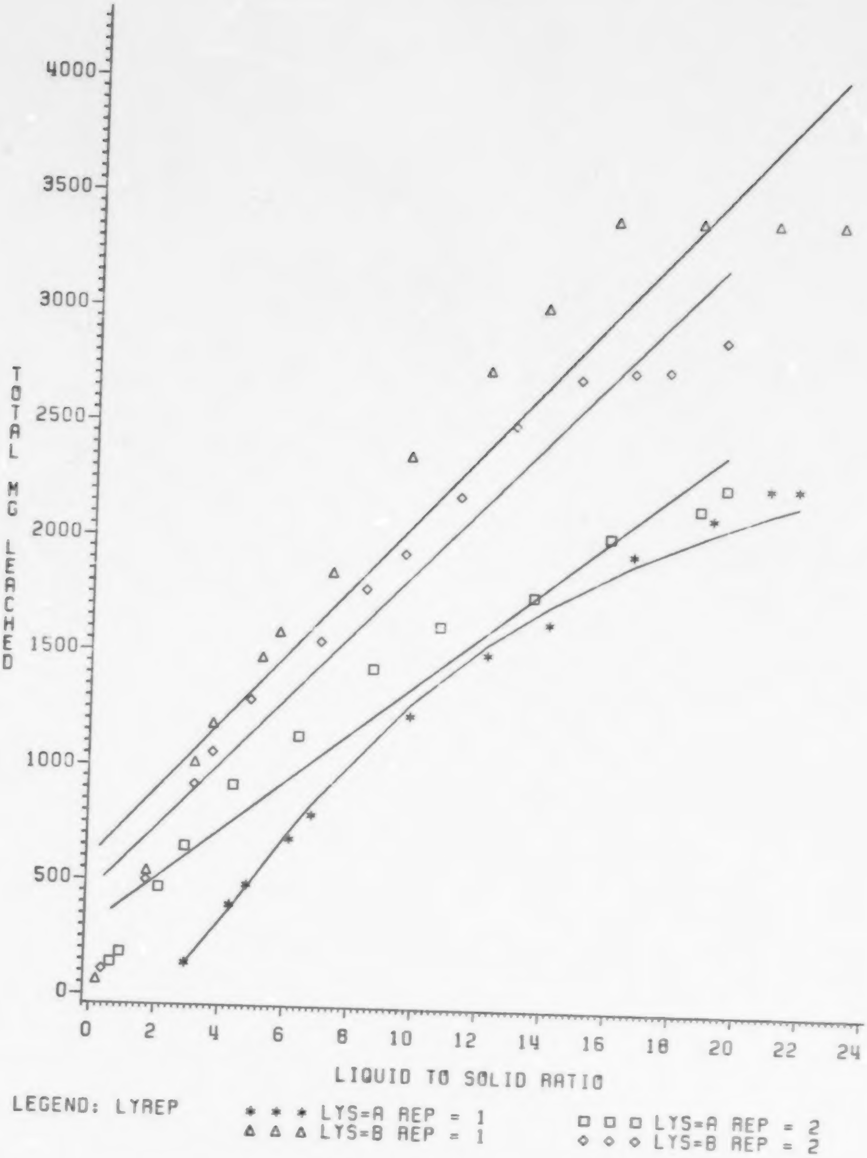

Fig. 56. Fitted accumulative leaching curves of trichloroethane from dichloroethylene still bottoms in Target, expressed in terms of liquid/solid ratios.

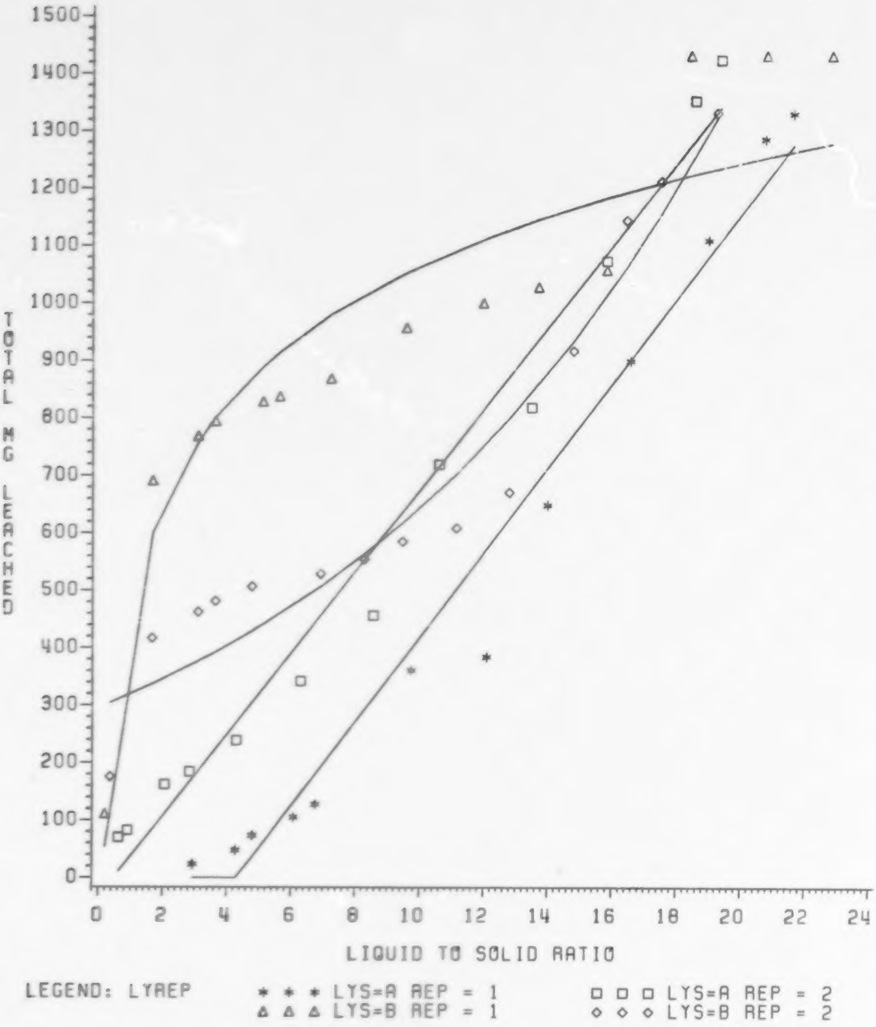

Fig. 57. Fitted accumulative leaching curves of trichloroethylene from dichloroethylene still bottoms in Target, expressed in terms of liquid/solid ratios.

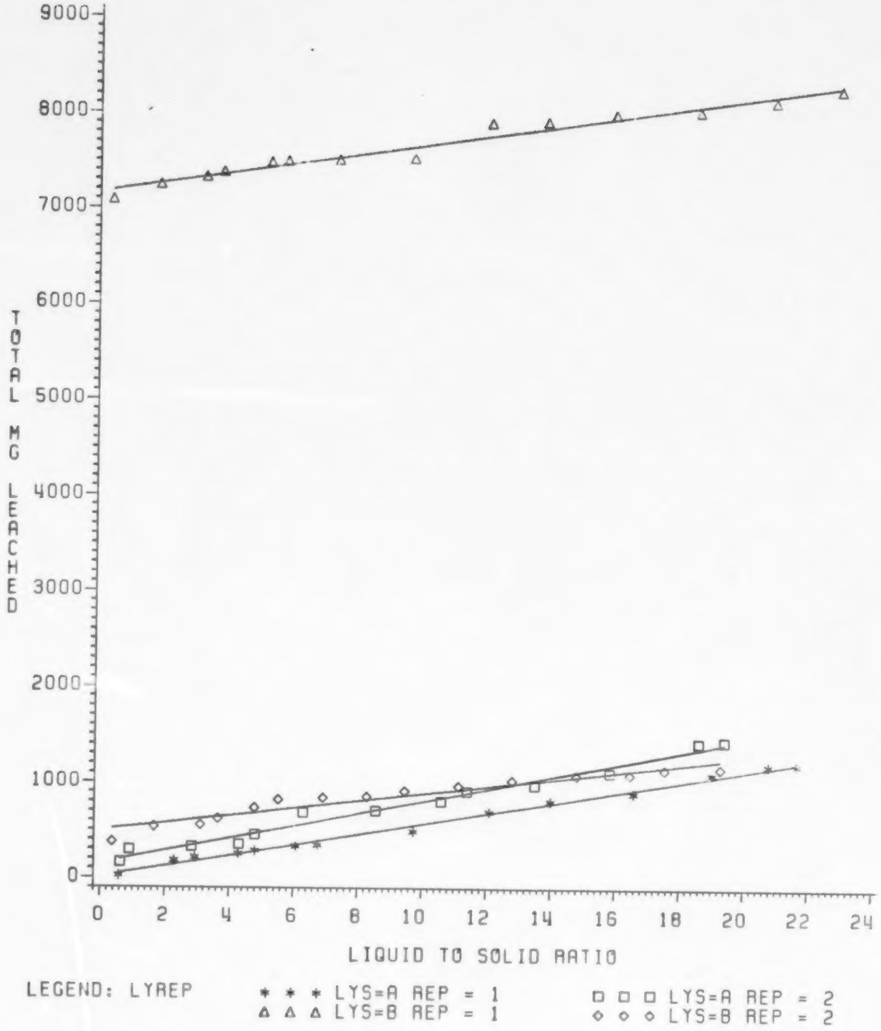

Fig. 58. Fitted accumulative leaching curves of hexachlorobutadiene from dichloroethylene still bottoms in Target, expressed in terms of liquid/solid ratios.

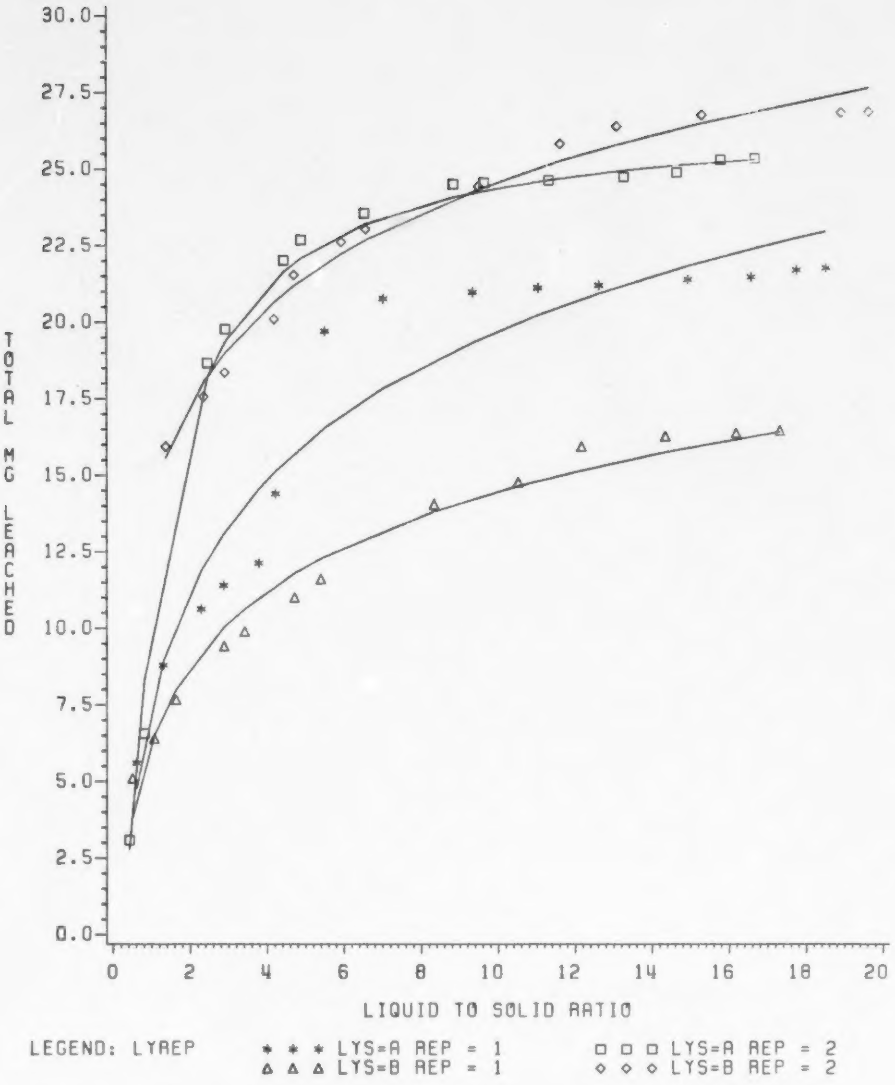

Fig. 59. Fitted accumulative leaching curves of barium from paint sludge in Target, expressed in terms of liquid/solid ratios.

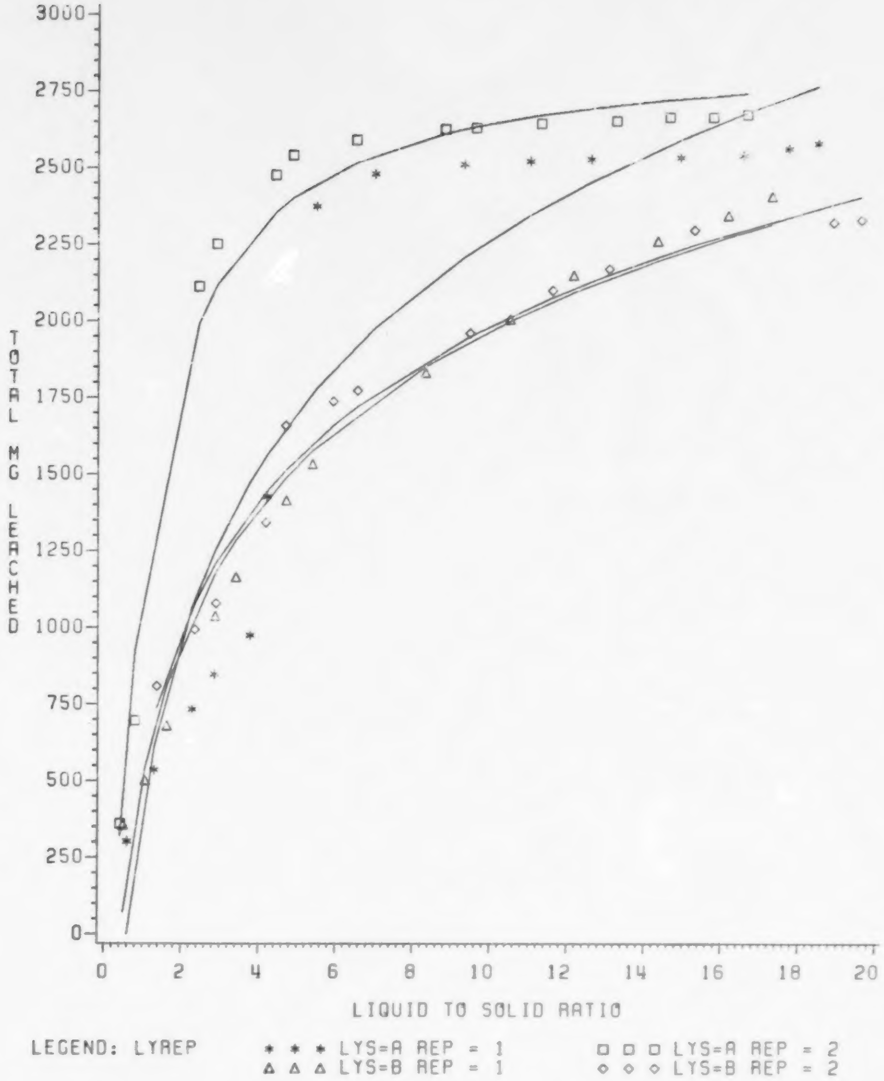

Fig. 60. Fitted accumulative leaching curves of zinc from paint sludge in Target, expressed in terms of liquid/solid ratios.

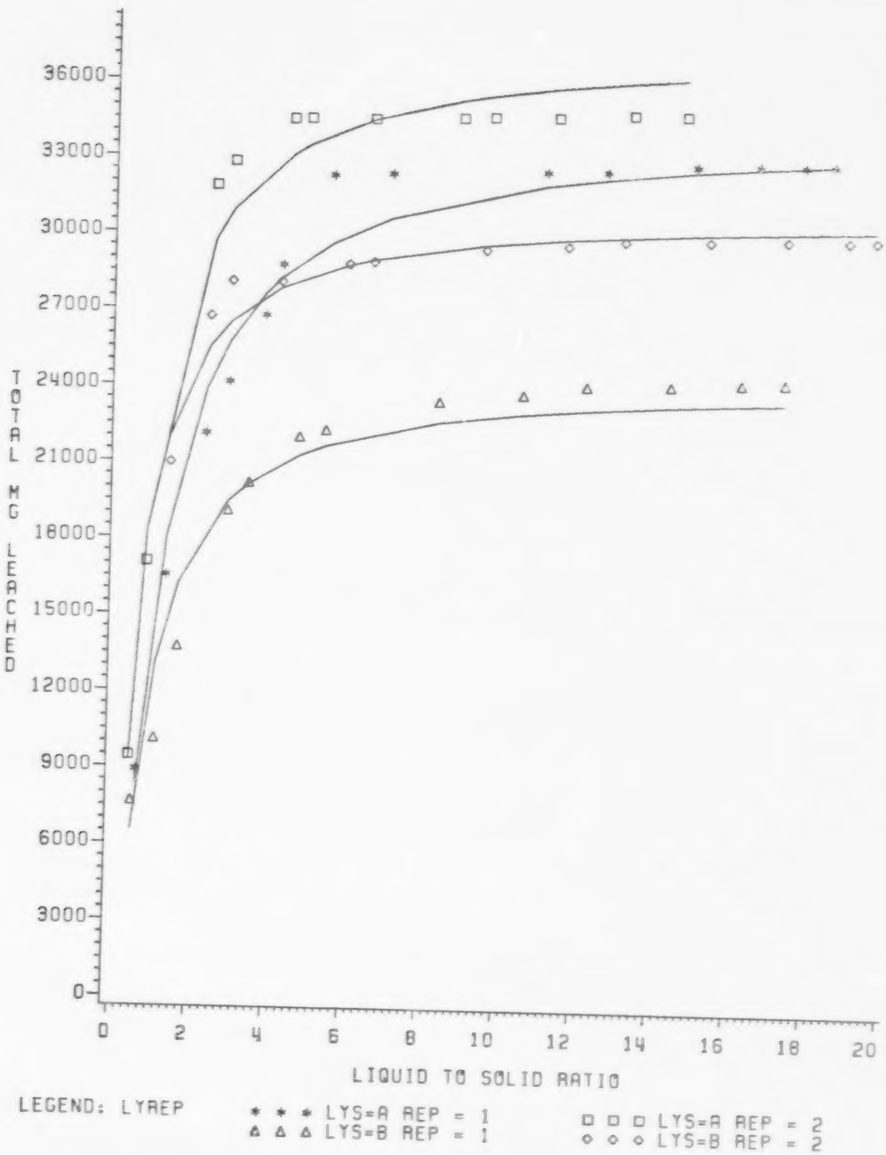

Fig. 61. Fitted accumulative leaching curves of ethoxyethanol from paint sludge in Target, expressed in terms of liquid/solid ratios.

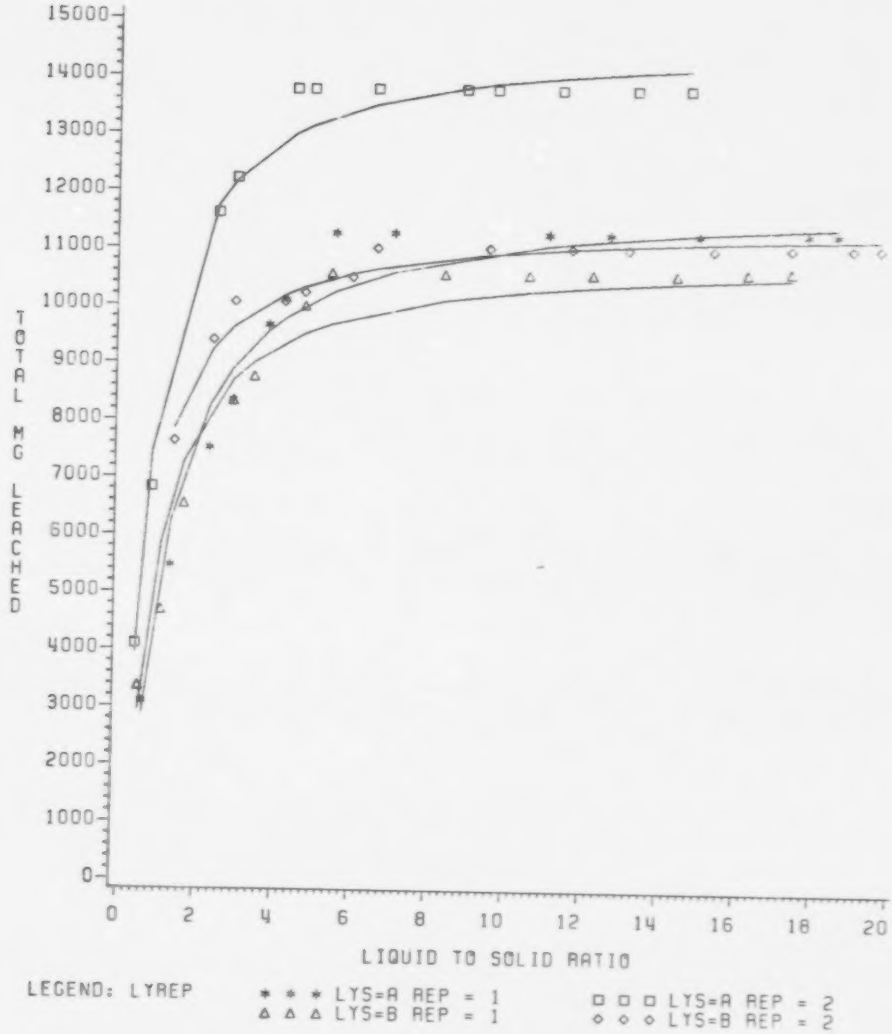

Fig. 62. Fitted accumulative leaching curves of ethoxyethyl acetate from paint sludge in Target, expressed in terms of liquid/solid ratios.

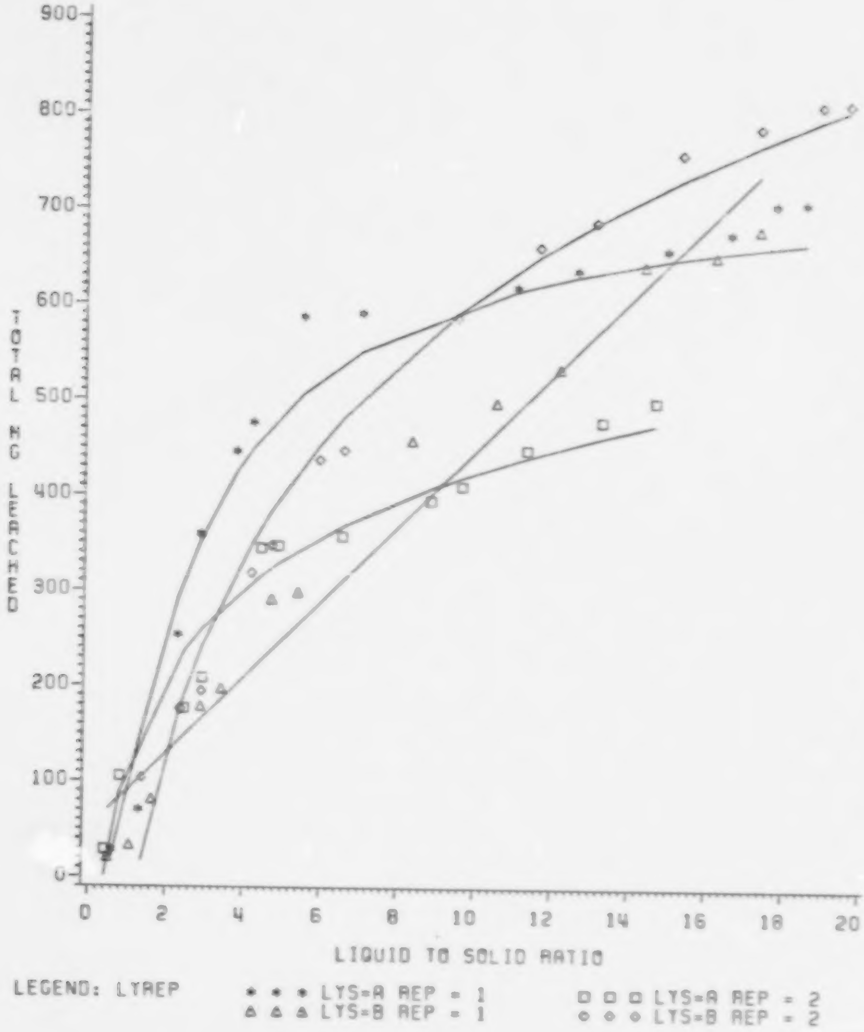

Fig. 63. Fitted accumulative leaching curves of toluene from paint sludge in Target, expressed in terms of liquid/solid ratios.

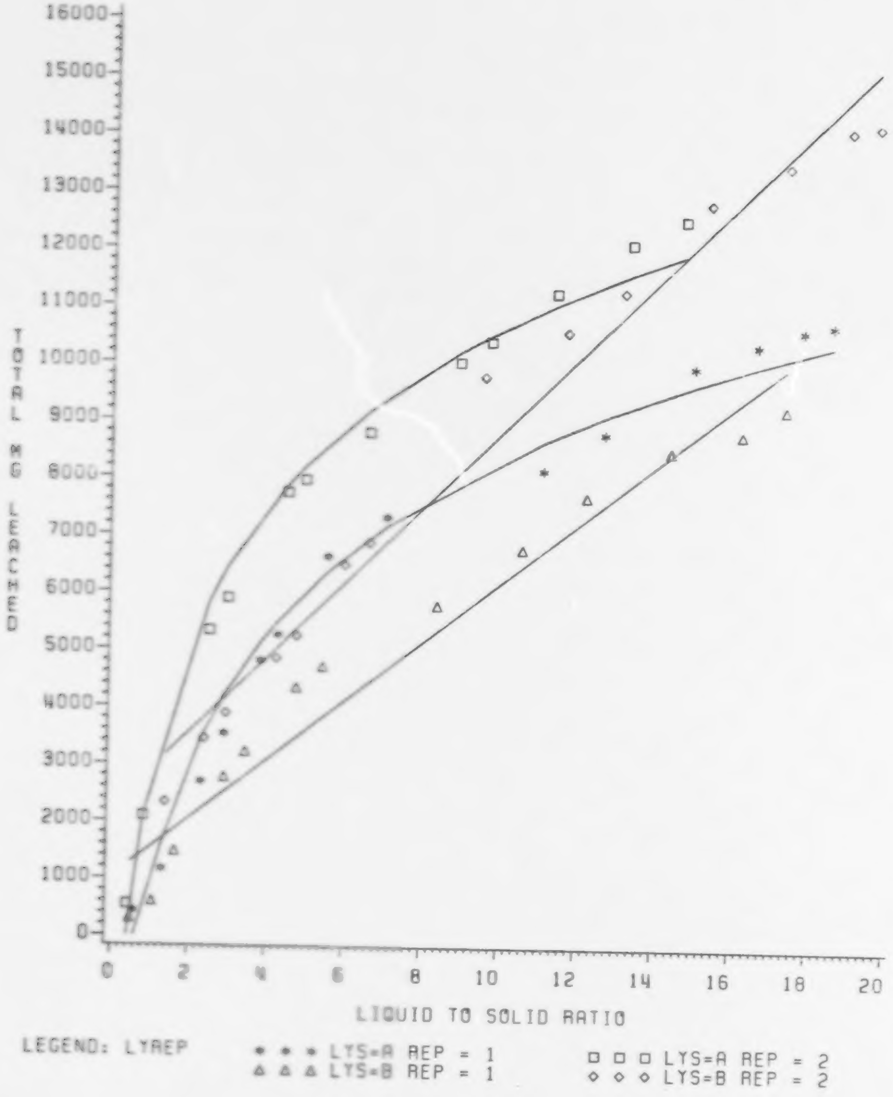

Fig. 64. Fitted accumulative leaching curves of xylenes from paint sludge in Target, expressed in terms of liquid/solid ratios.

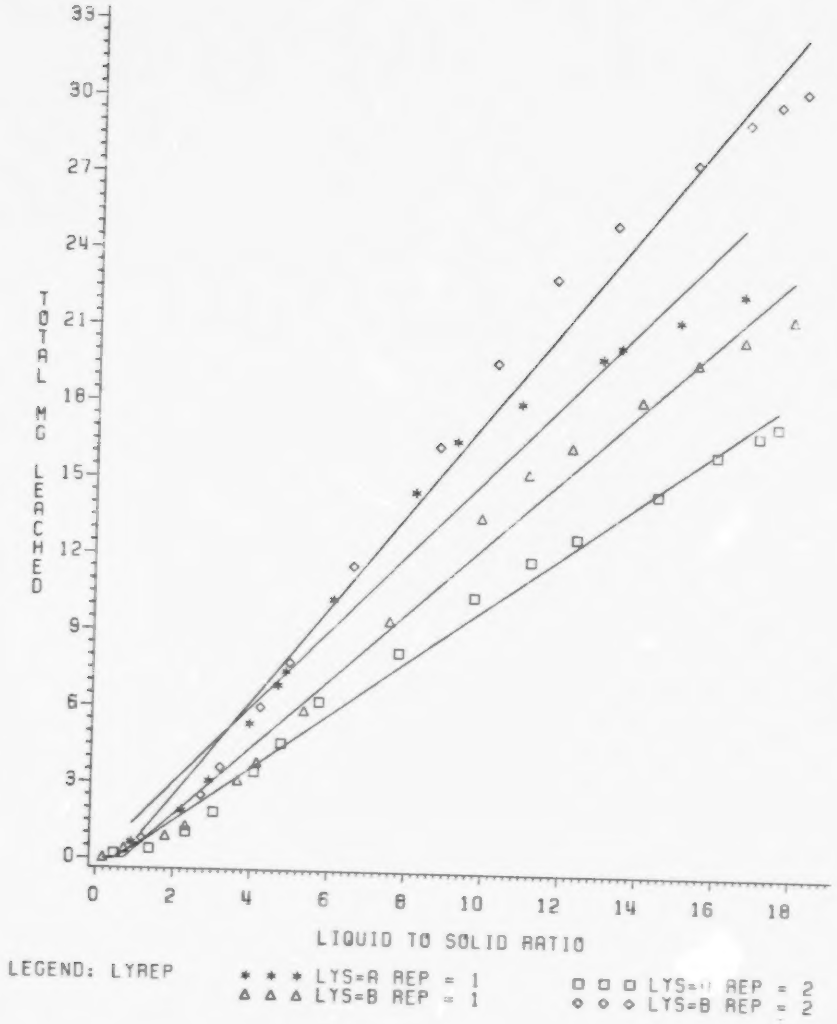

Fig. 65. Fitted accumulative leaching curves of barium from electroplating waste in Target, expressed in terms of liquid/solid ratios.

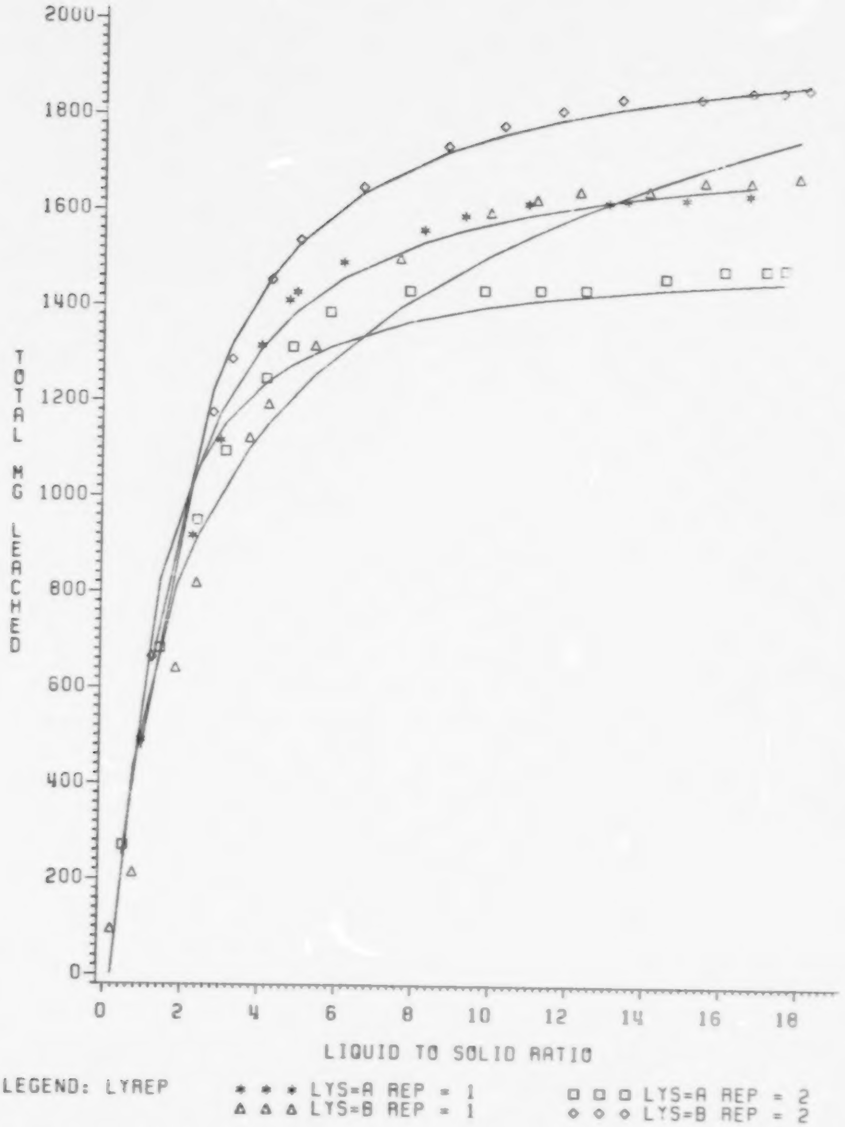

Fig. 66. Fitted accumulative leaching curves of boron from electroplating waste in Target, expressed in terms of liquid/solid ratios.

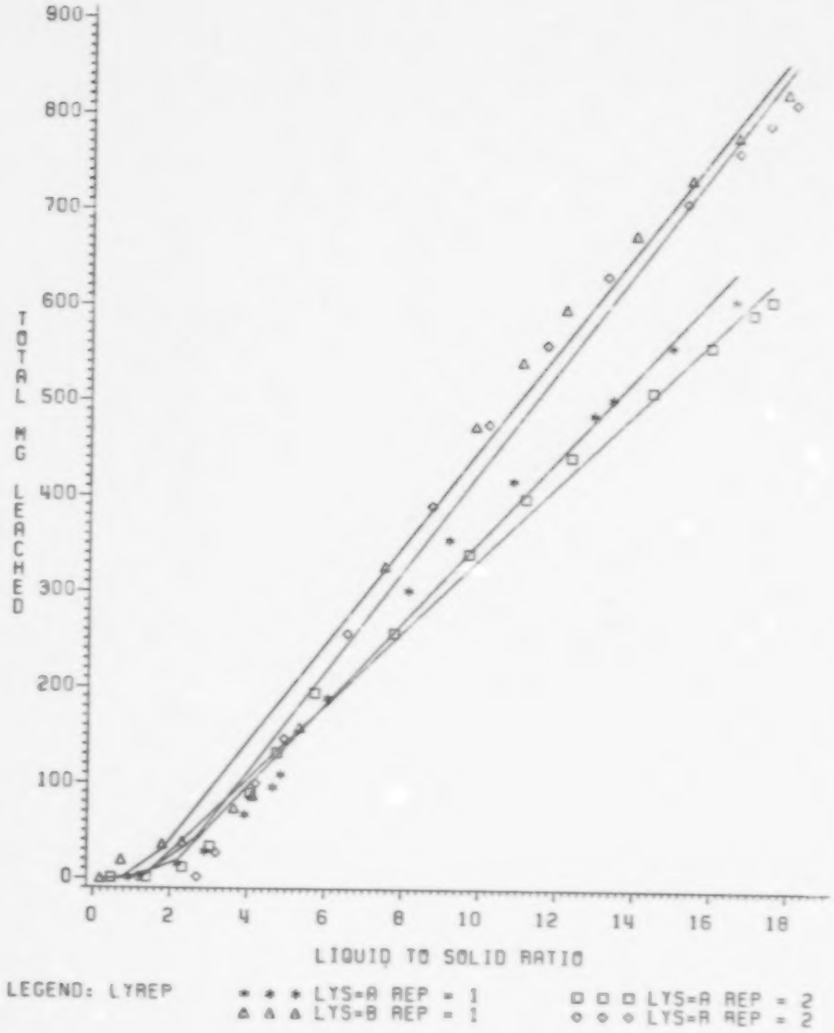

Fig. 67. Fitted accumulative leaching curves of manganese from electroplating waste in Target, expressed in terms of liquid/solid ratios.

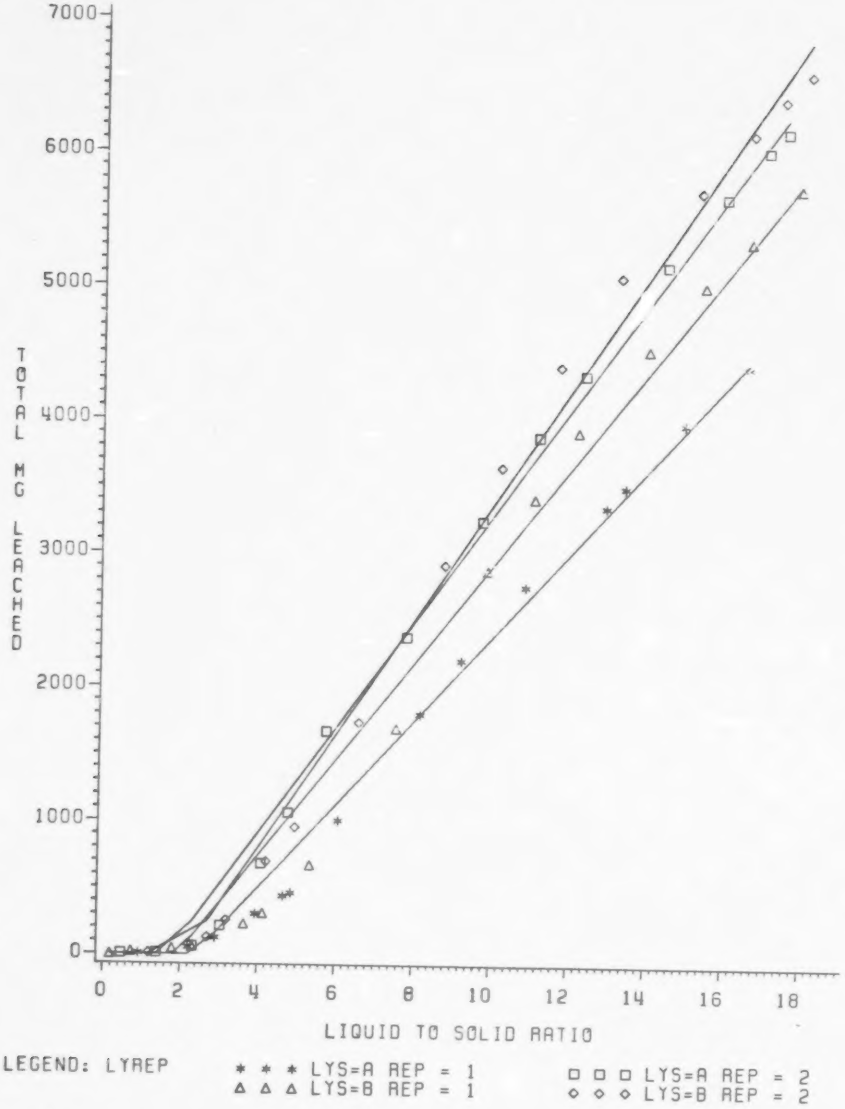

Fig. 68. Fitted accumulative leaching curves of nickel from electroplating waste in Target, expressed in terms of liquid/solid ratios.

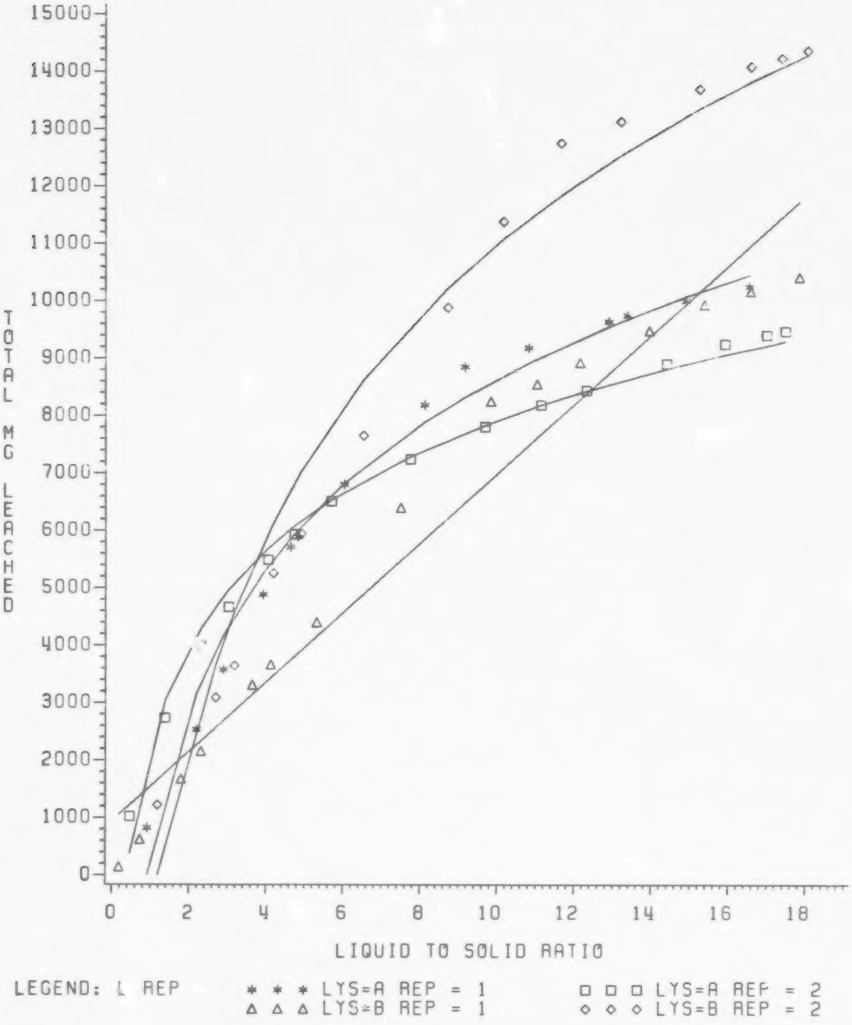

Fig. 69. Fitted accumulative leaching curves of potassium from electroplating waste in Target, expressed in terms of liquid/solid ratios.

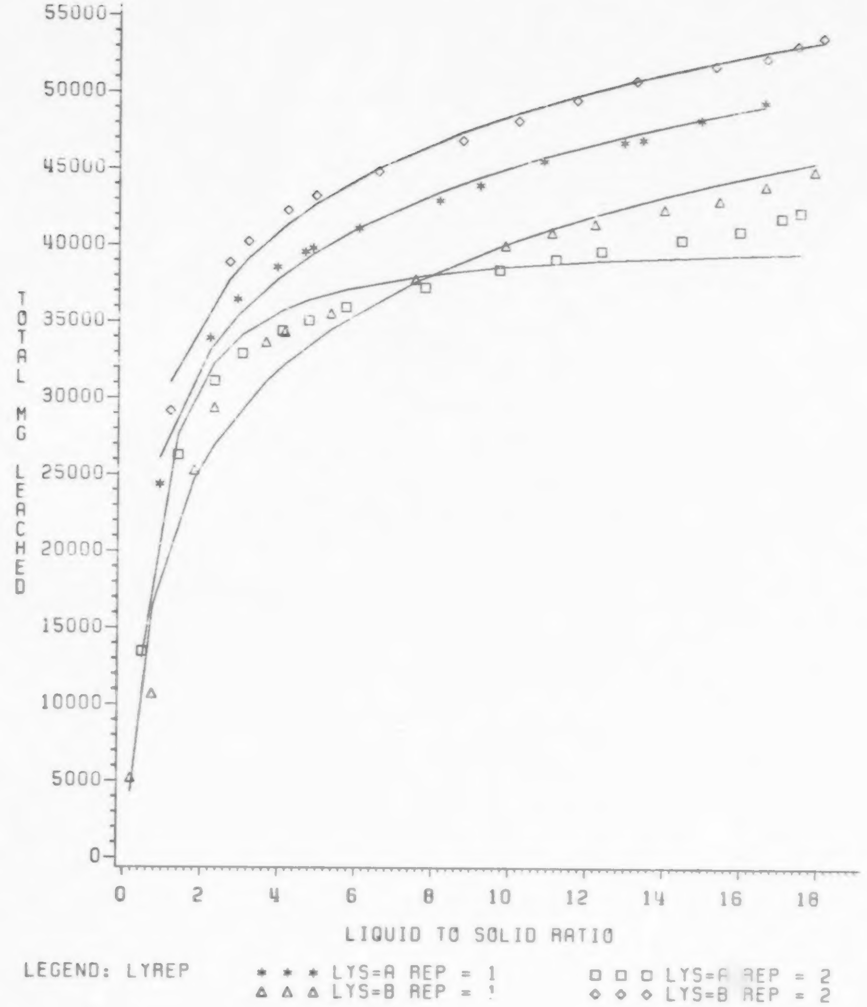

Fig. 70. Fitted accumulative leaching curves of sodium from electroplating waste in Target, expressed in terms of liquid/solid ratios.

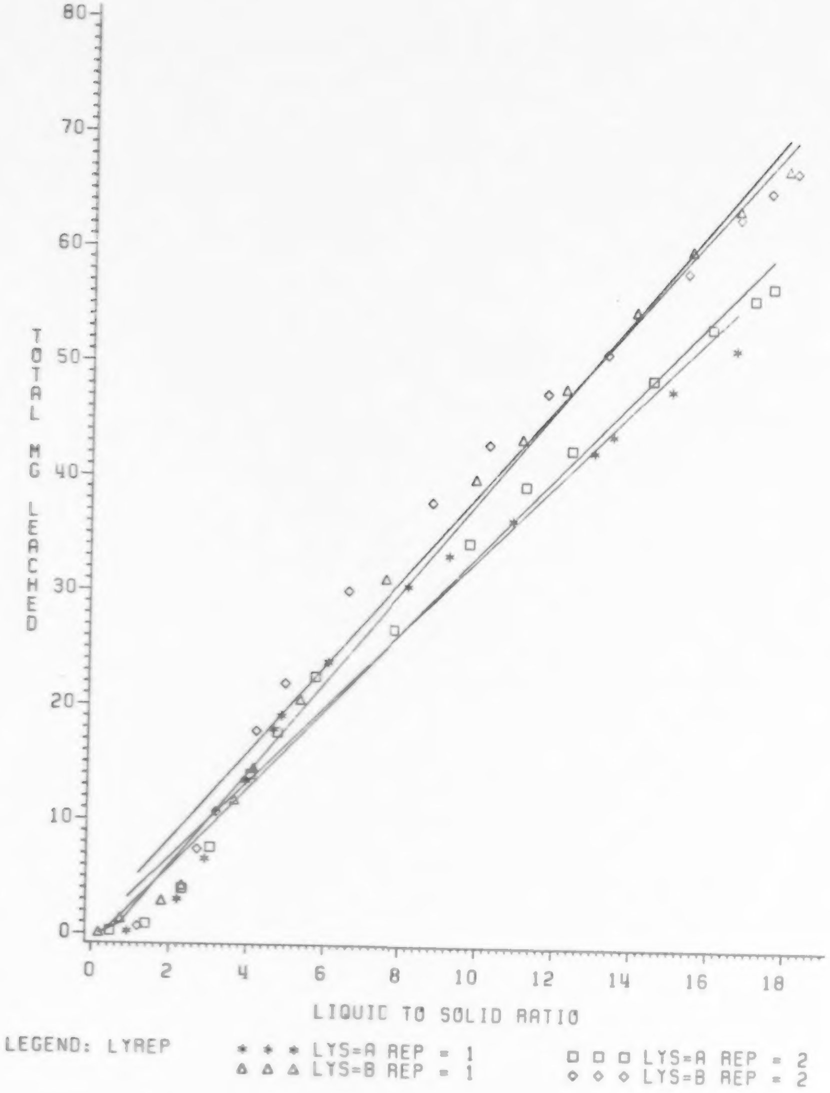

Fig. 71. Fitted accumulative leaching curves of strontium from electroplating waste in Target, expressed in terms of liquid/solid ratios.

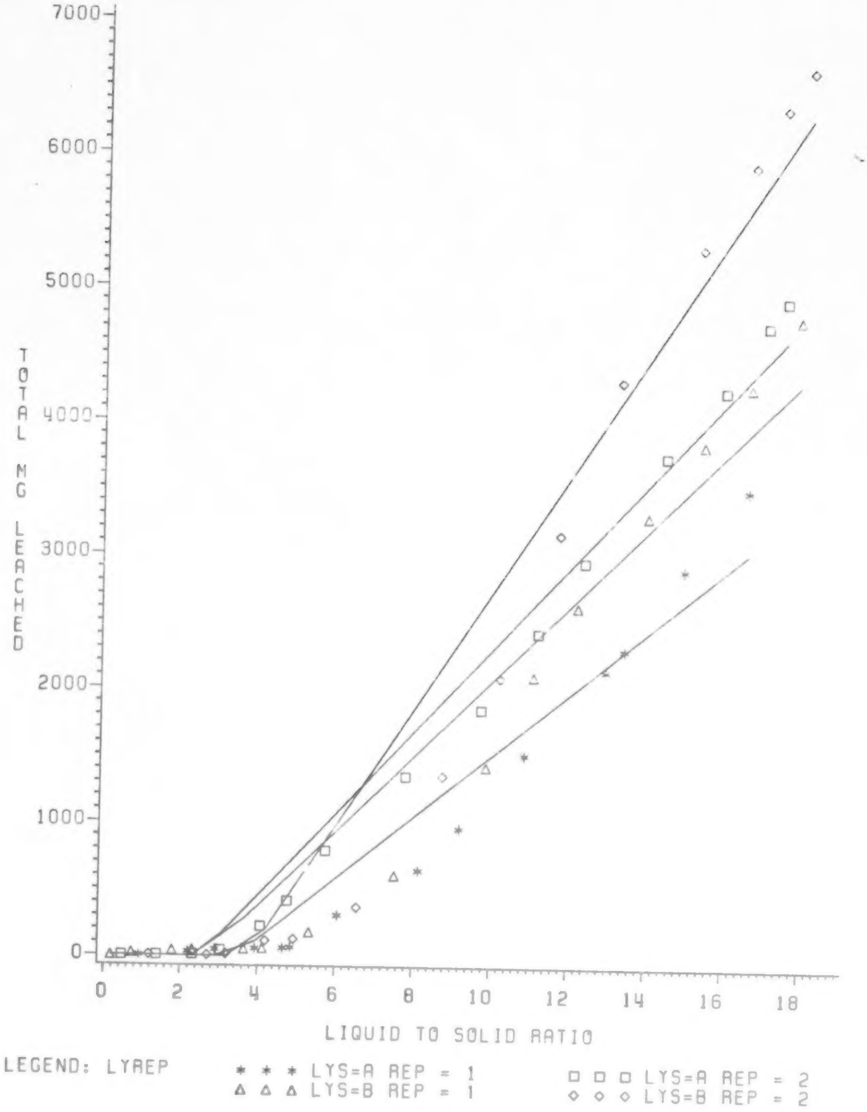

Fig. 72. Fitted accumulative leaching curves of zinc from electroplating waste in Target, expressed in terms of liquid/solid ratios.

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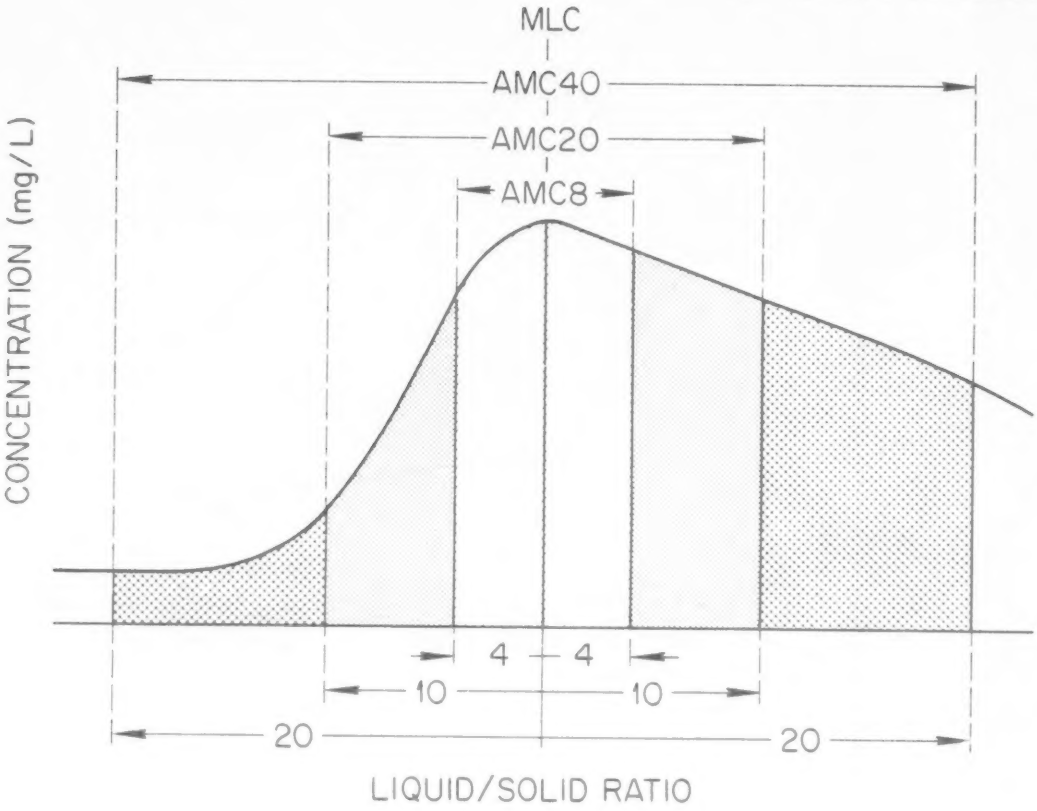

Fig. 73. An illustration of how AMC target concentrations are determined.

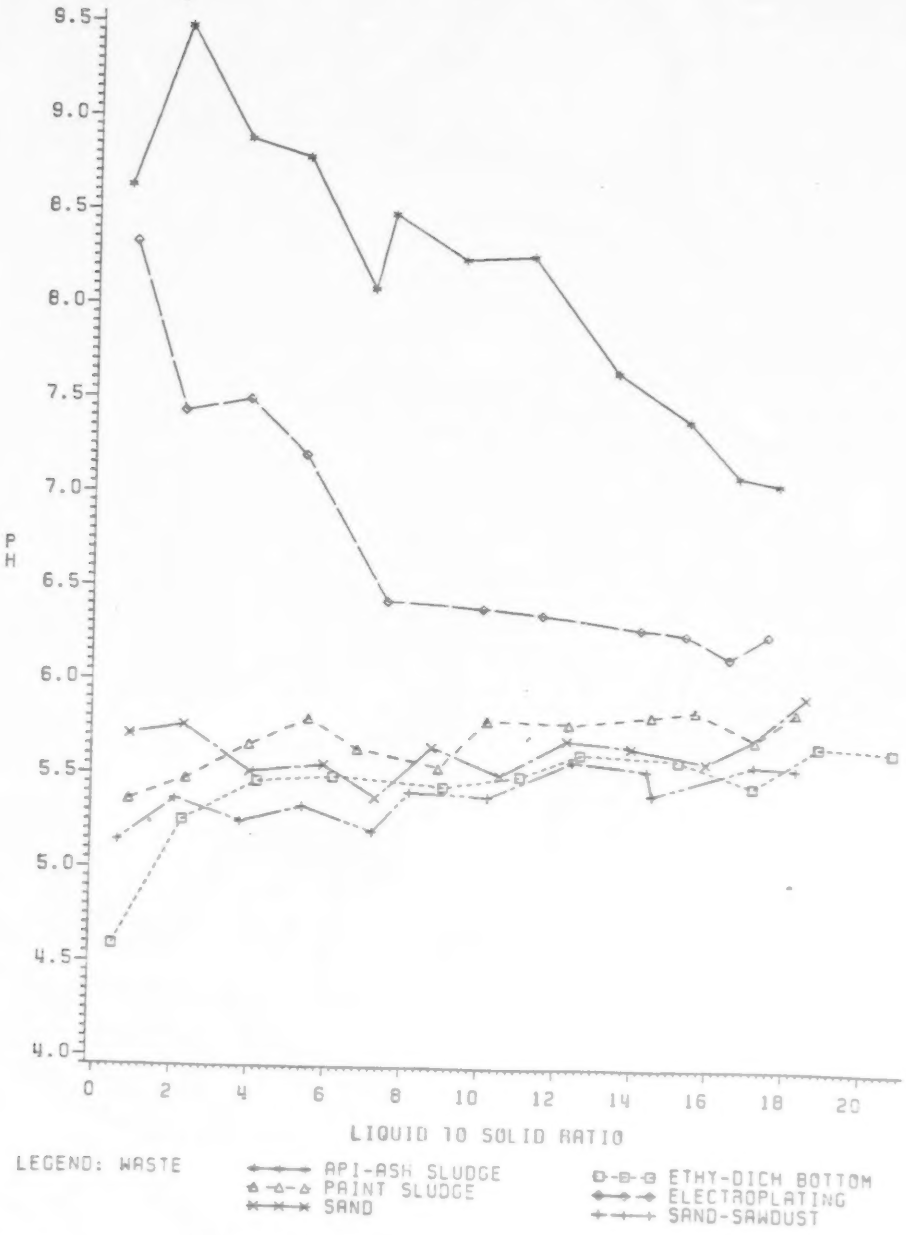

Fig. 74. Leachate pH of Target as a function of liquid/solid ratio.

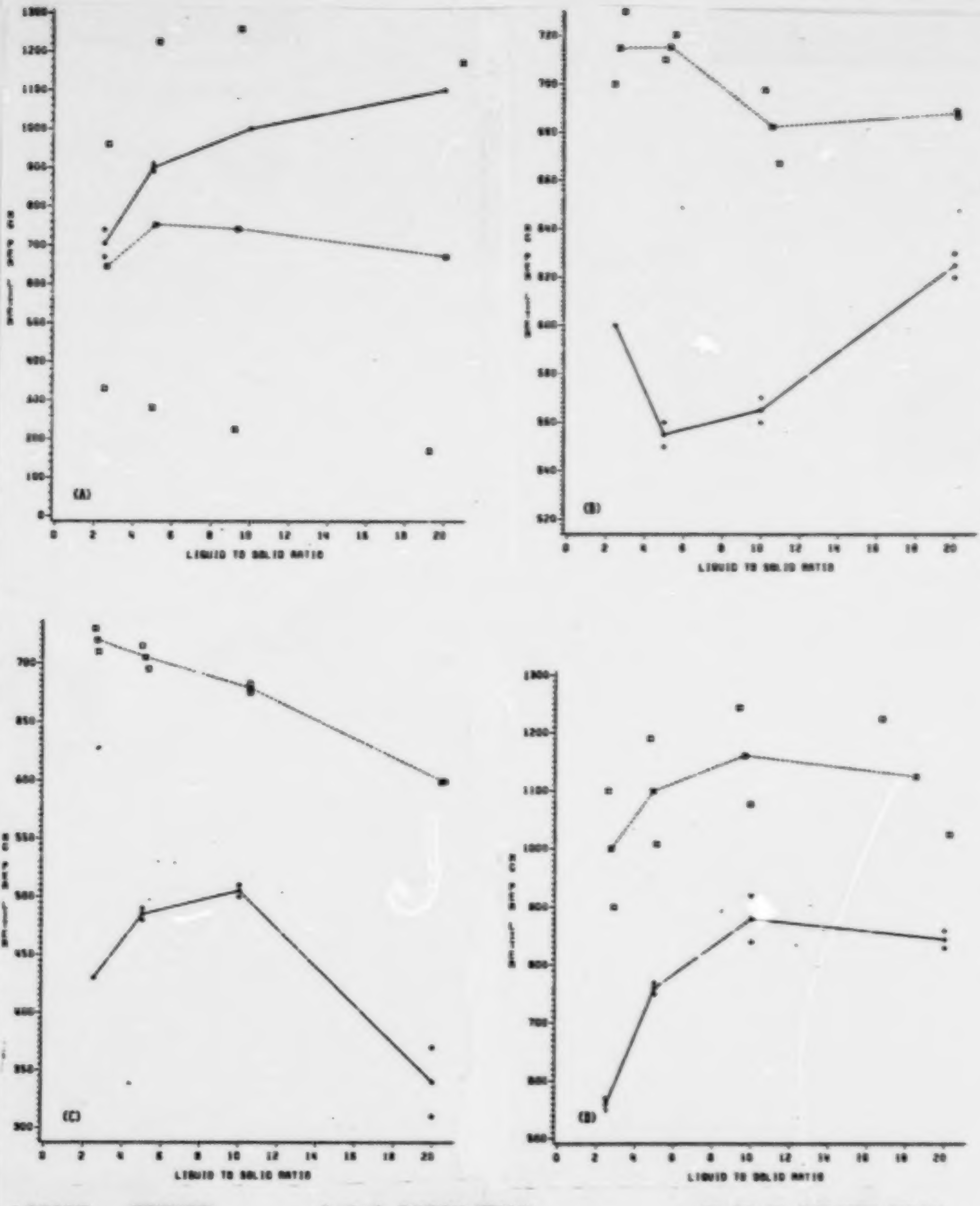

Fig. 75. Comparison of rotary-batch and upflow-column extraction methods for calcium in API/incinerator ash by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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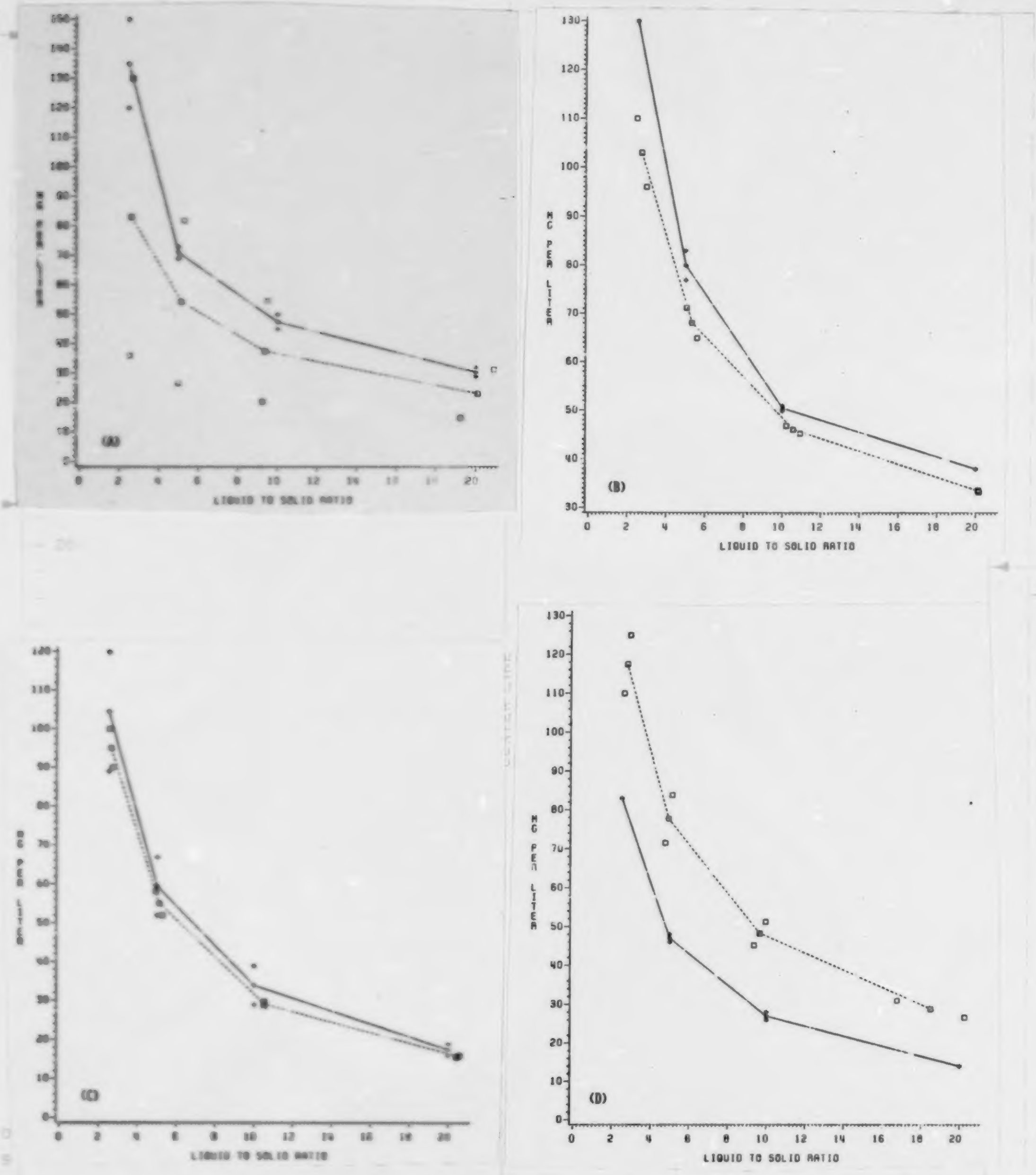

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Fig. 76. Comparison of rotary-batch and upflow-column extraction methods for chromium in API/incinerator ash by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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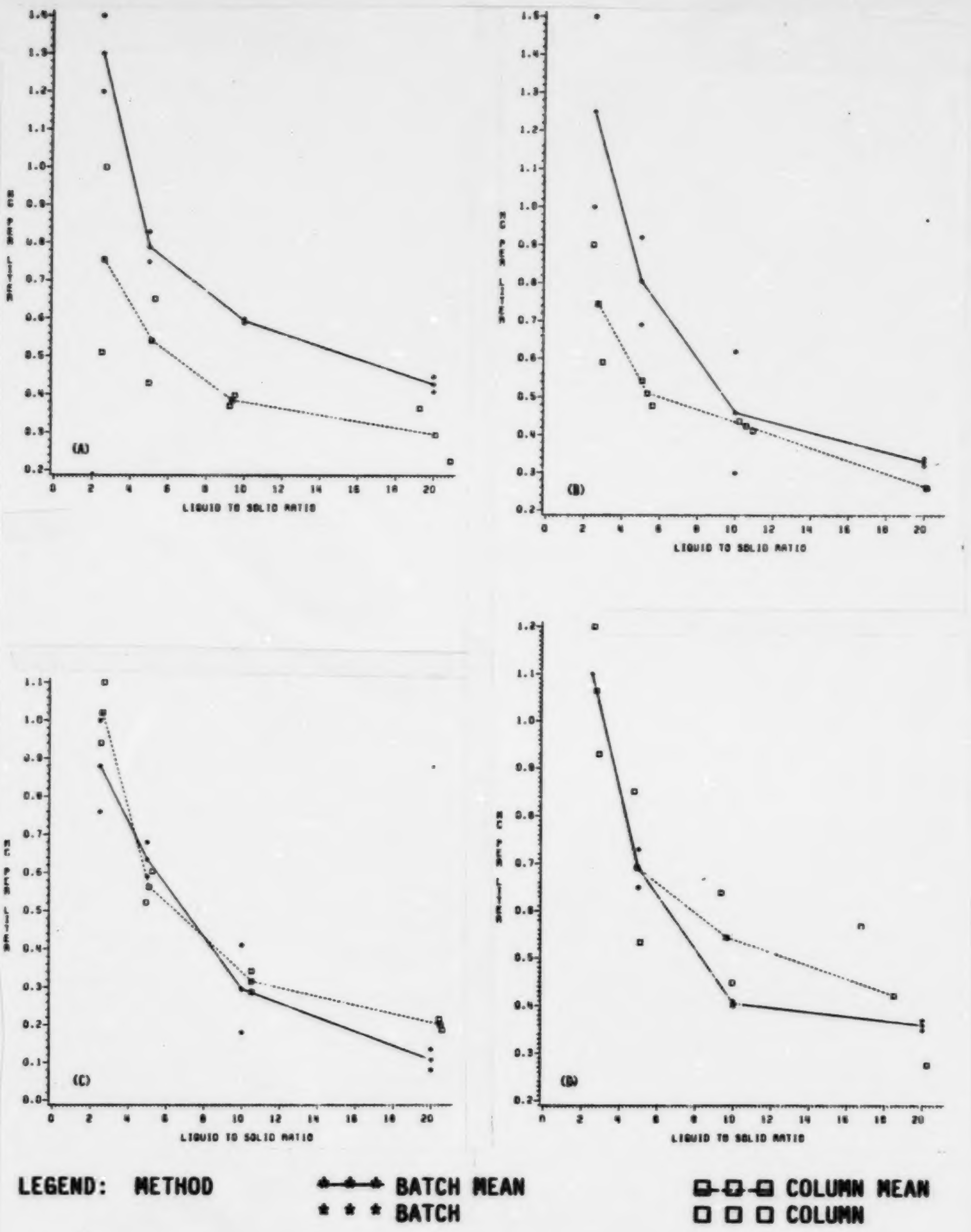

Fig. 77. Comparison of rotary-batch and upflow-column extraction methods for molybdenum in API/incinerator ash by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

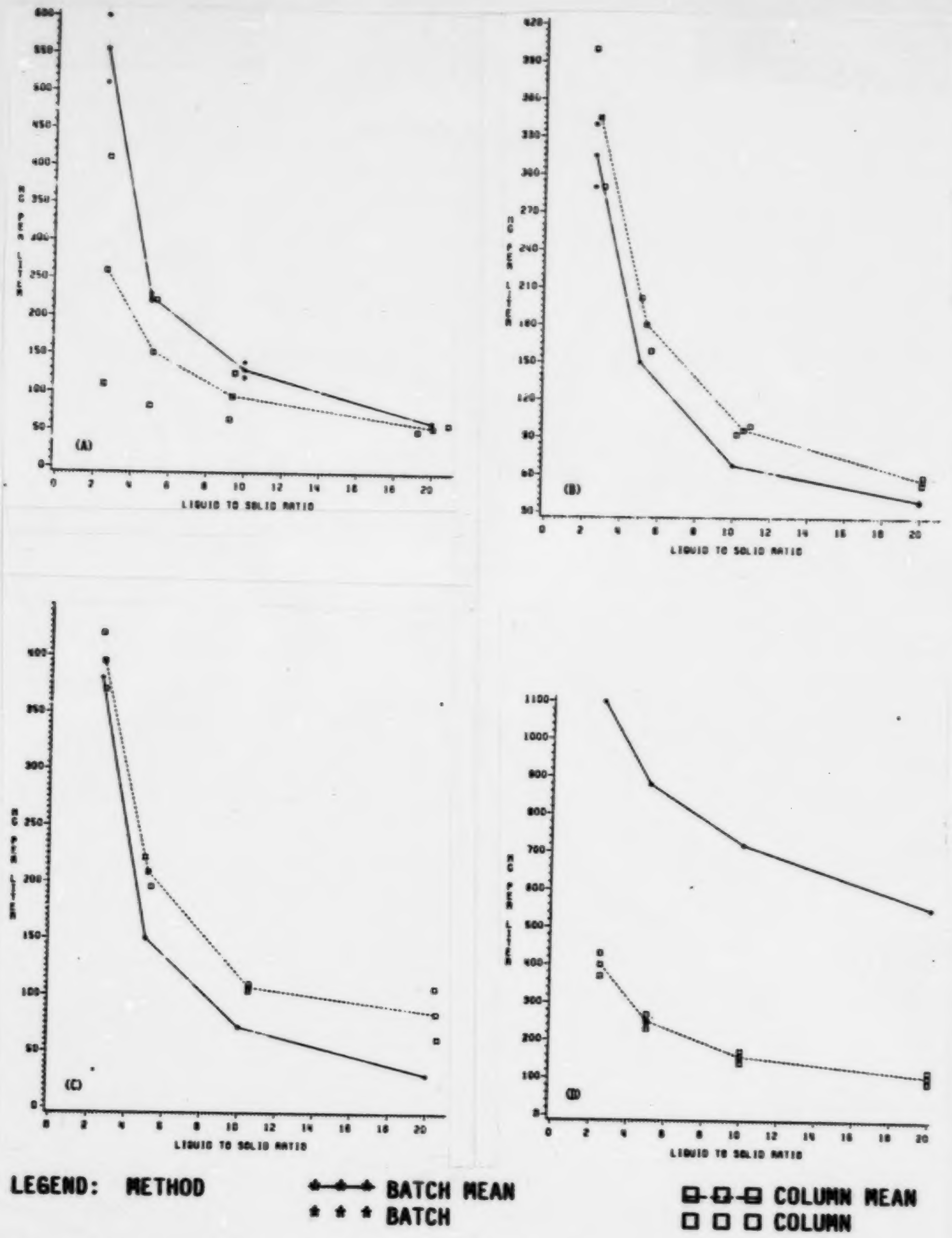

Fig. 78. Comparison of rotary-batch and upflow-column extraction methods for potassium in API/incinerator ash by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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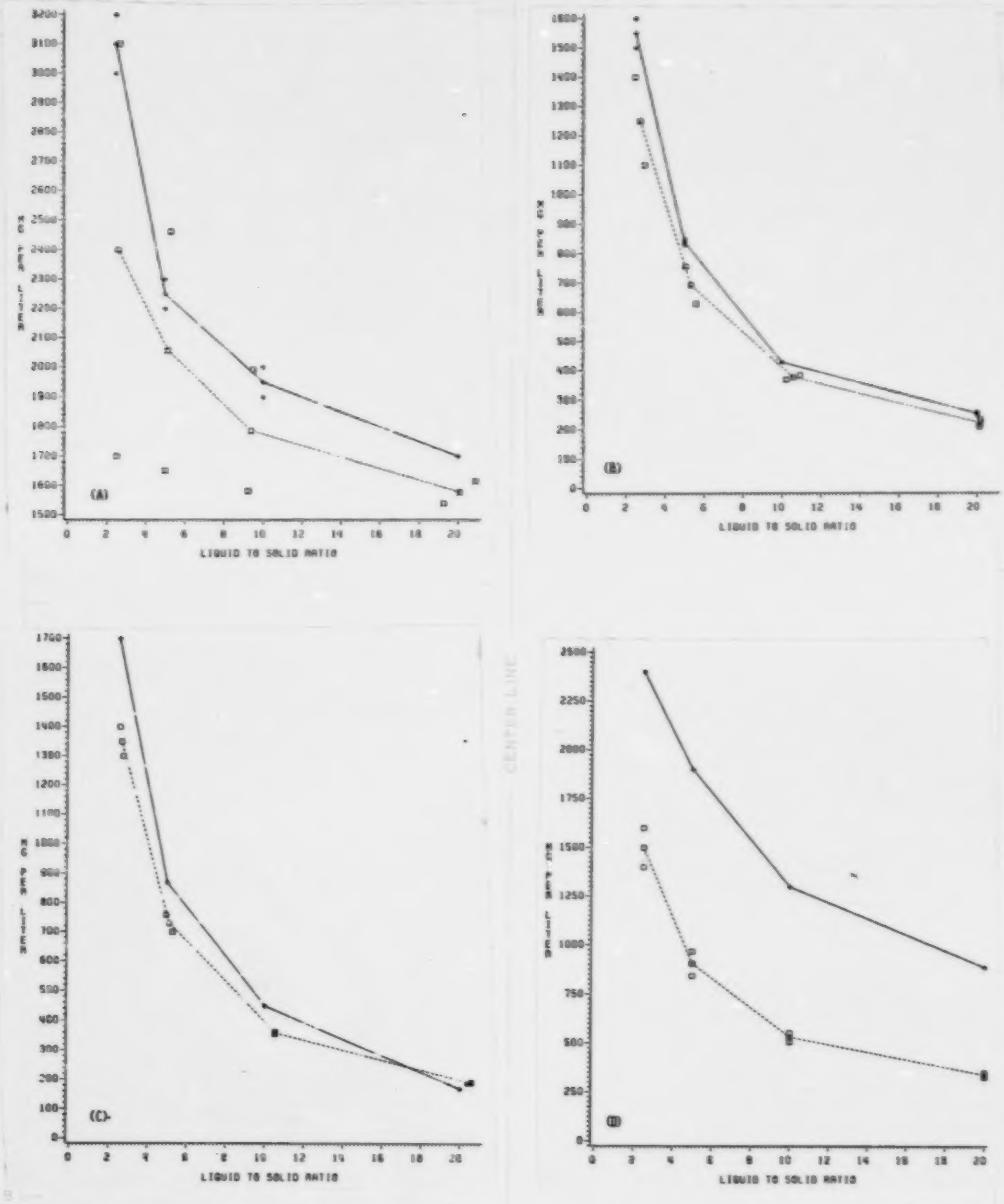

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Fig. 79. Comparison of rotary-batch and upflow-column extraction methods for sodium in API/incinerator ash by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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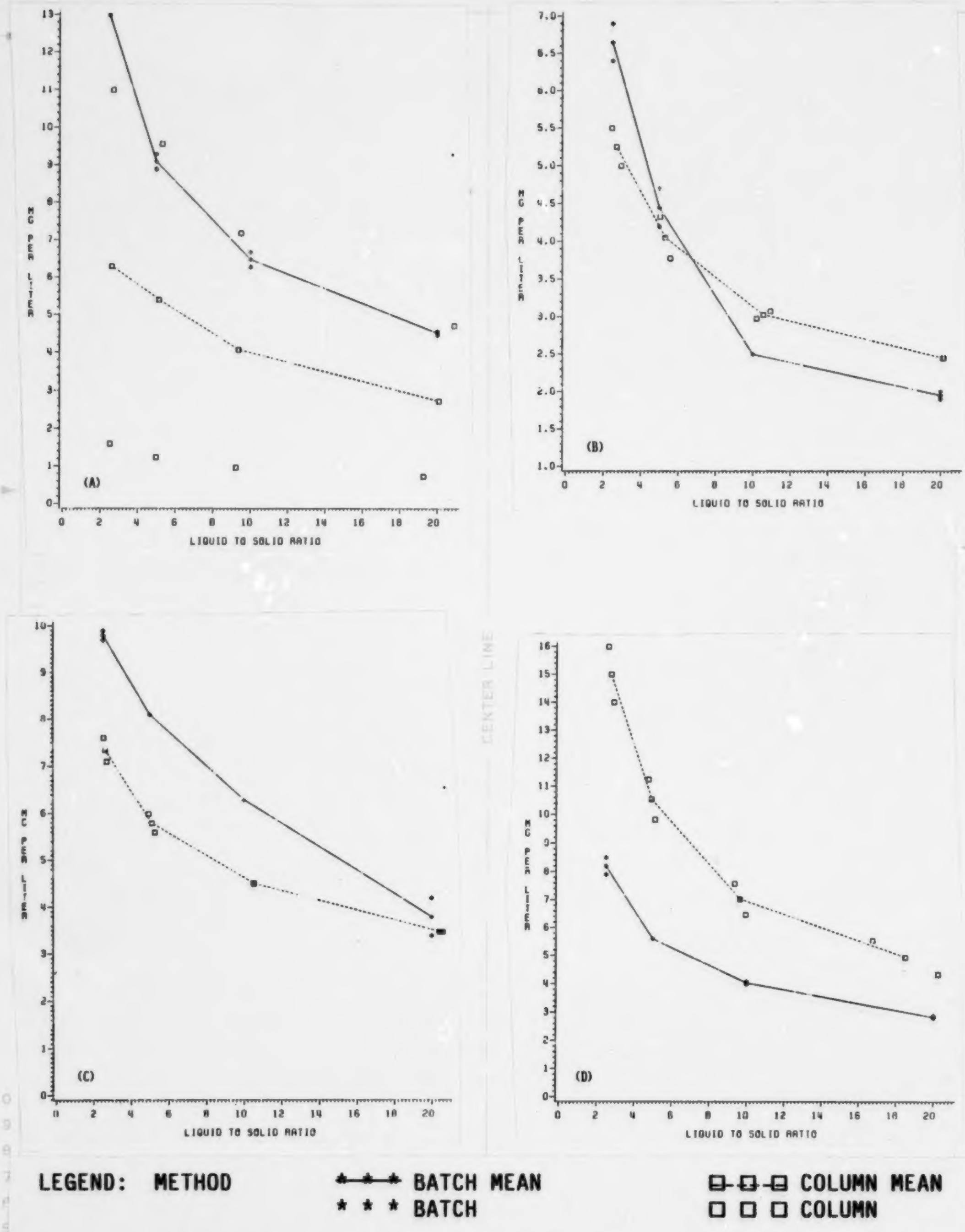

Fig. 80. Comparison of rotary-batch and upflow-column extraction methods for strontium in API/incinerator ash by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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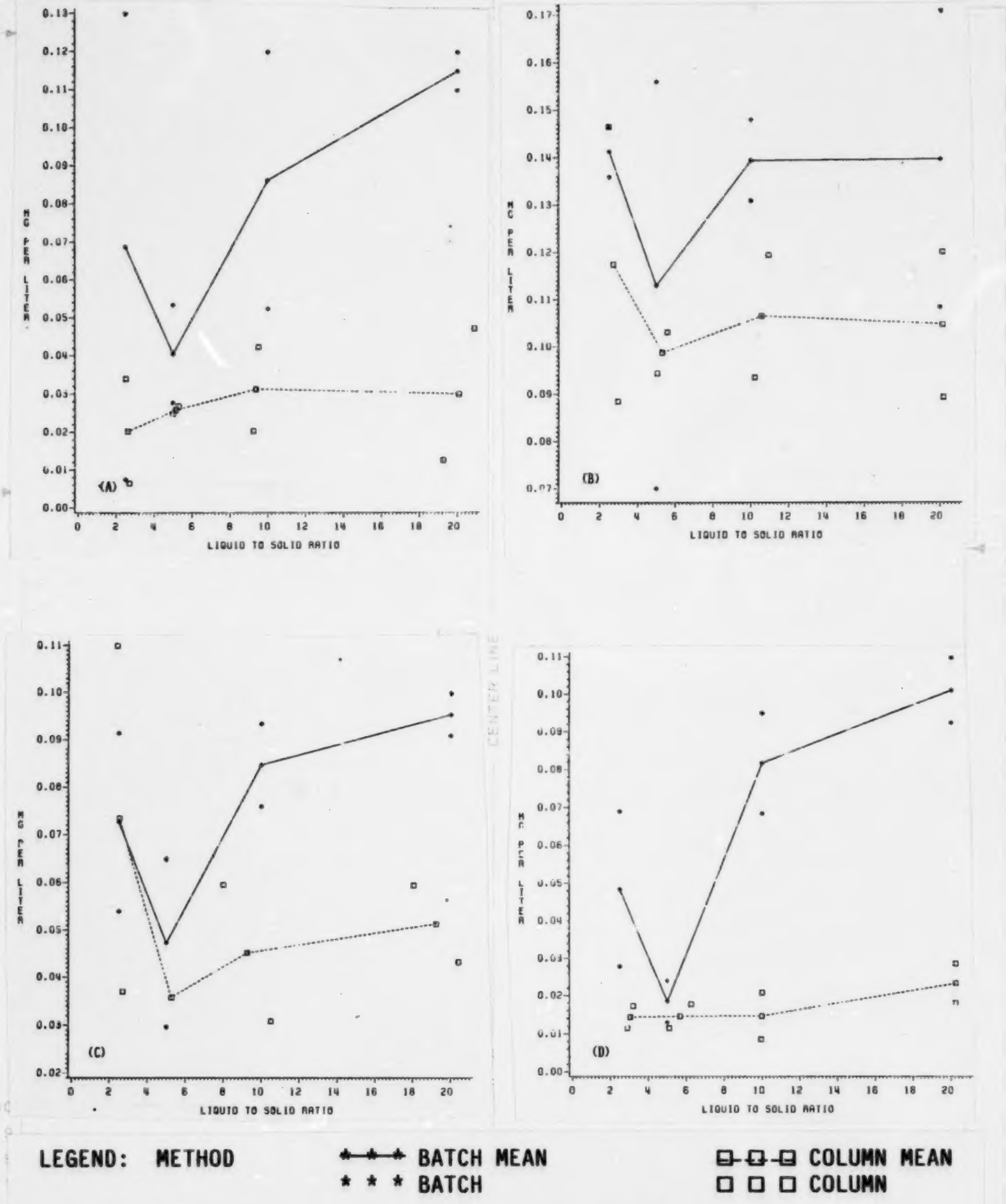

Fig. 81. Comparison of rotary-batch and upflow-column extraction methods for naphthalene in API/incinerator ash by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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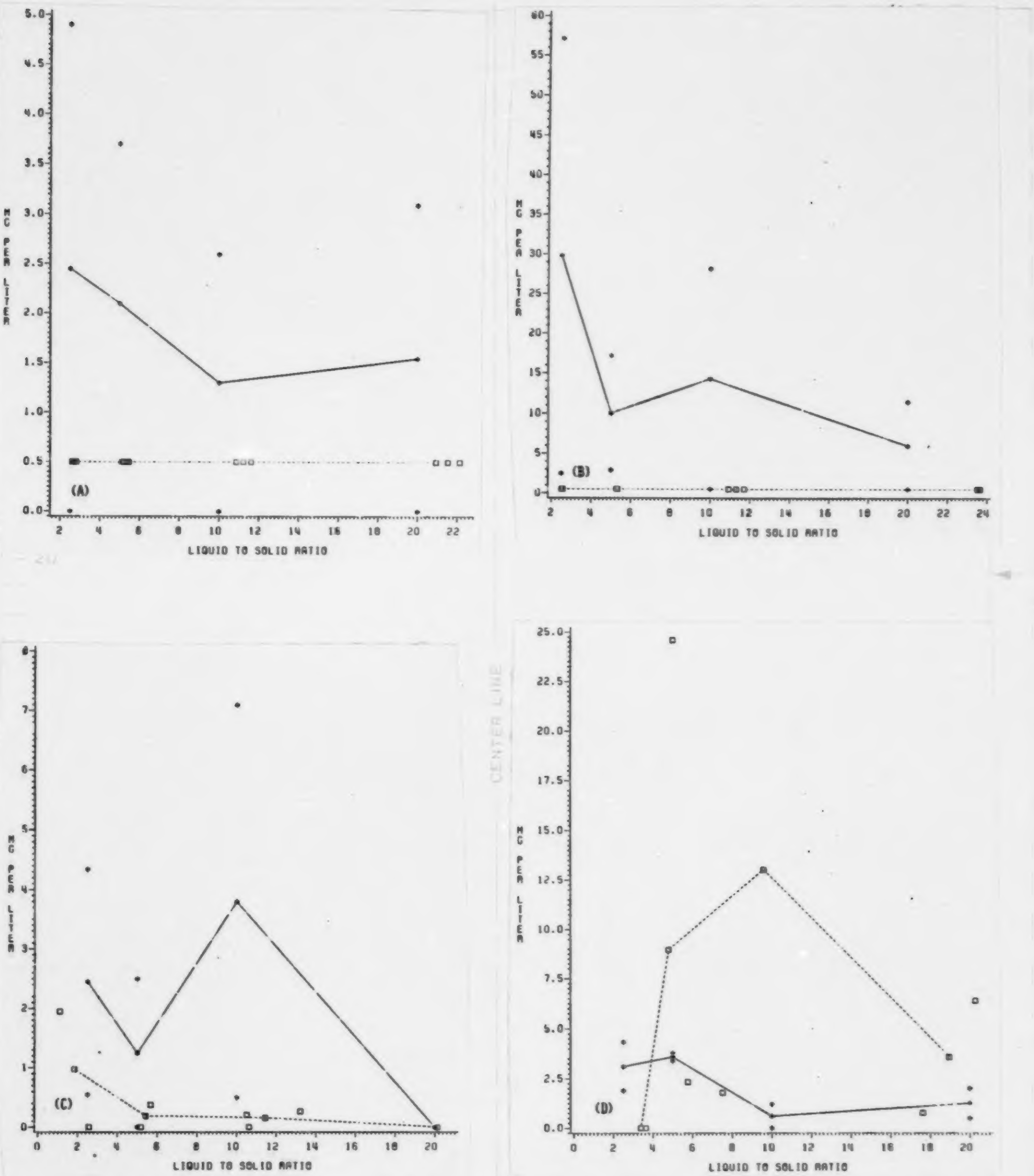

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✱ ✱ ✱ **BATCH** ☐ ☐ ☐ **COLUMN**

Fig. 82. Comparison of rotary-batch and upflow-column extraction methods for dichloroethane in dichloroethylene still bottoms by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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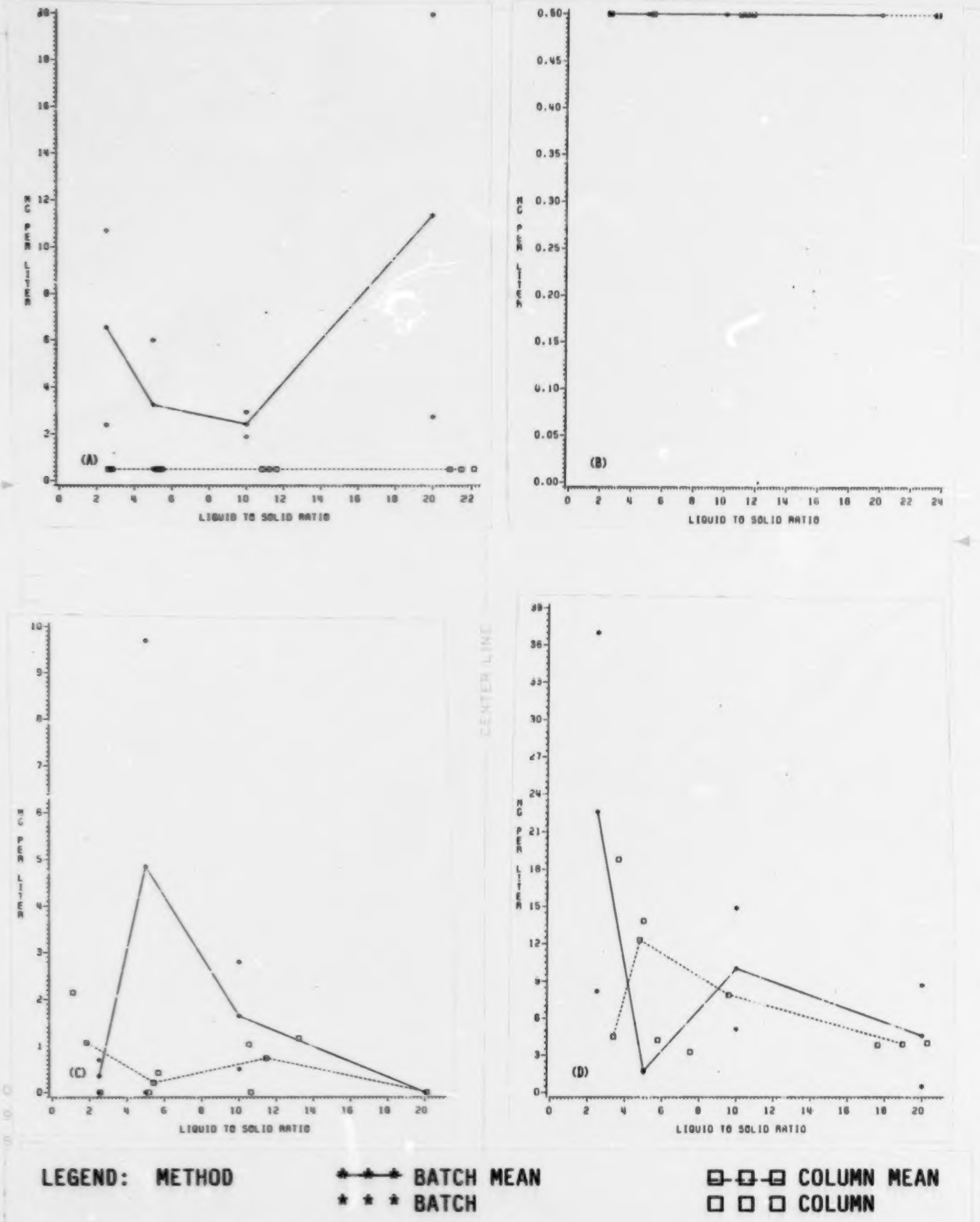

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Fig. 83. Comparison of rotary-batch and upflow-column extraction methods for trichloroethane in dichloroethylene still bottoms by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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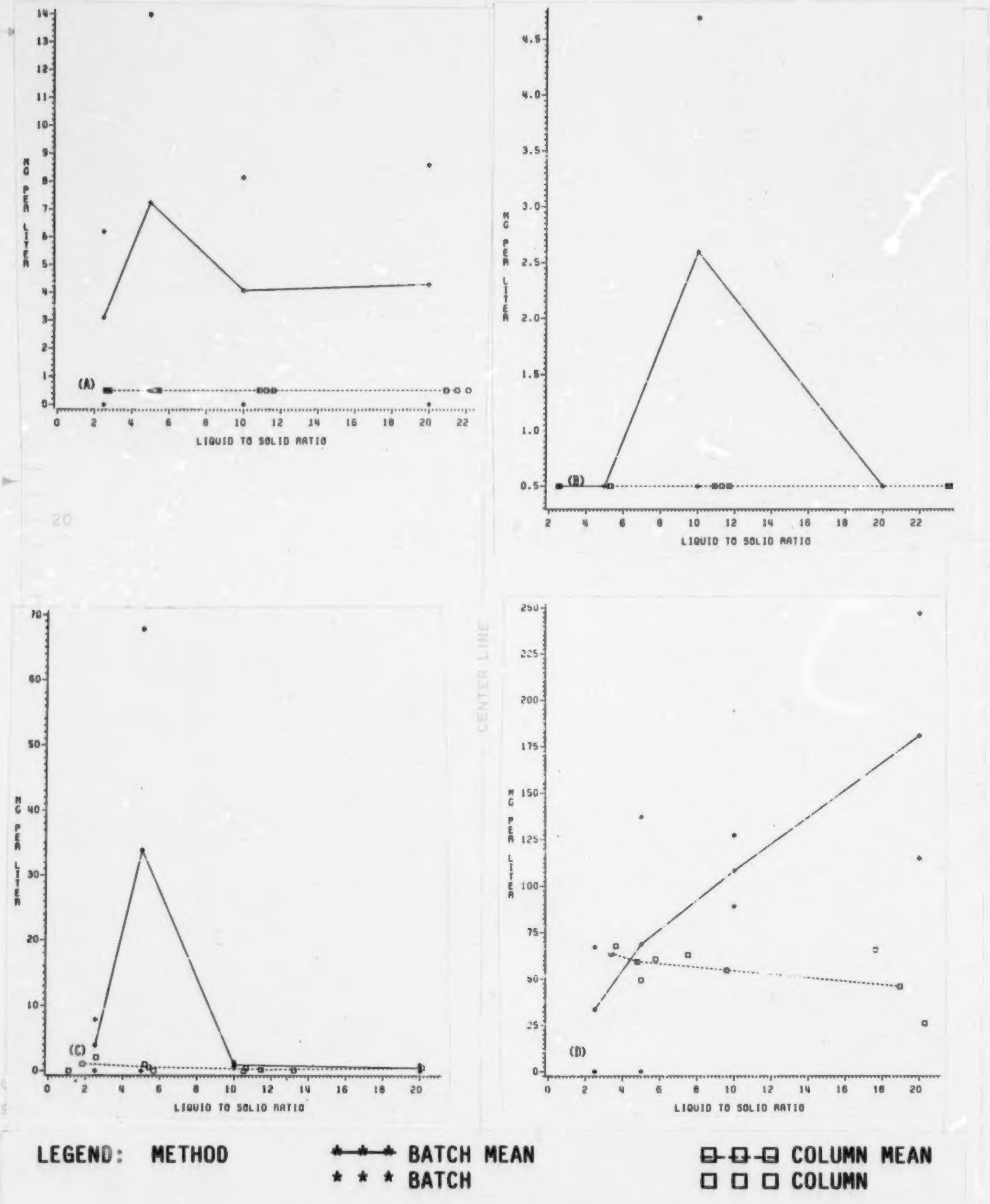
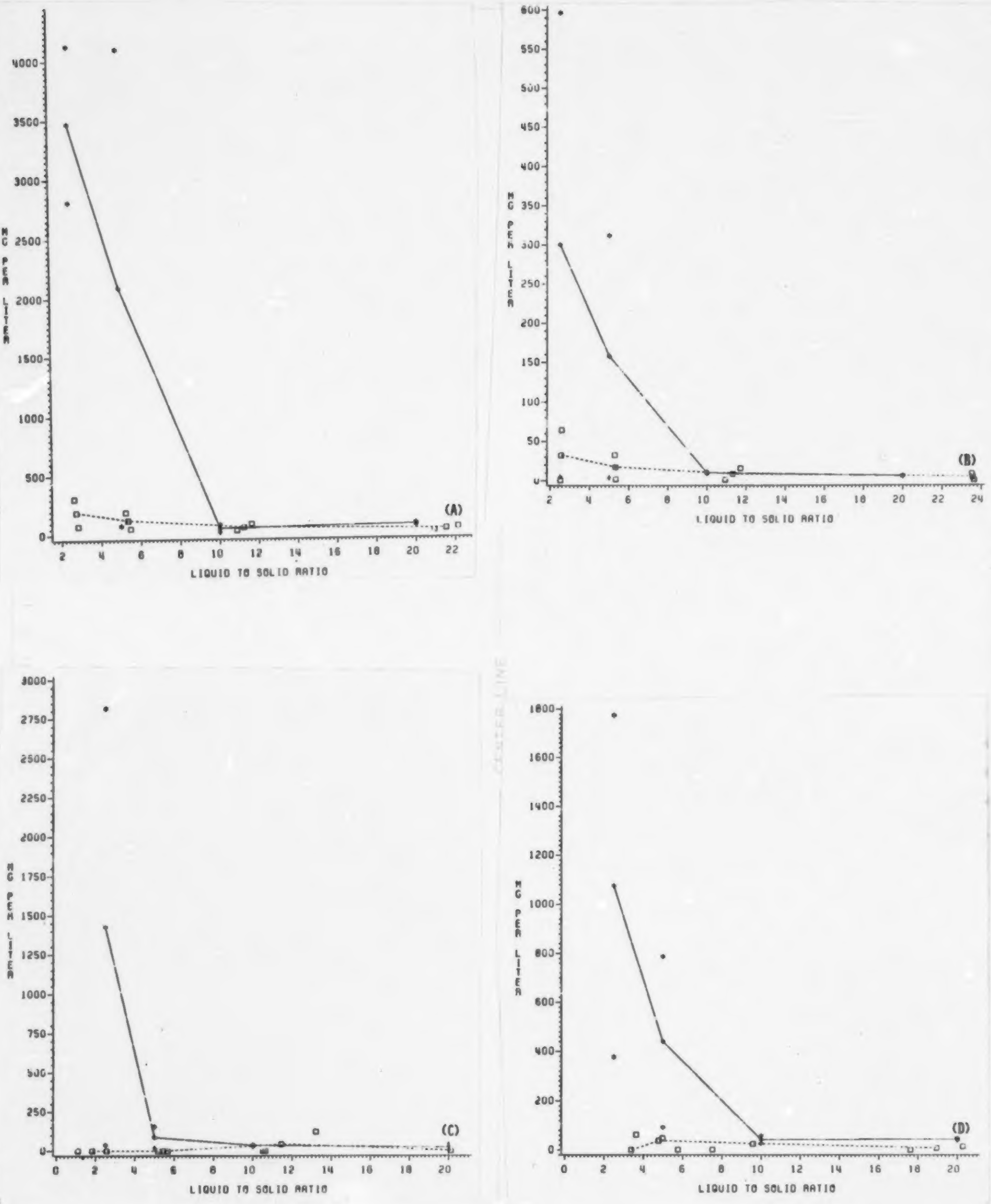


Fig. 84. Comparison of rotary-batch and upflow-column extraction methods for trichloroethylene in dichloroethylene still bottoms by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.



LEGEND: METHOD ✱—✱—✱ BATCH MEAN ☐-☐-☐ COLUMN MEAN
✱ ✱ ✱ BATCH ☐ ☐ ☐ COLUMN

Fig. 85. Comparison of rotary-batch and upflow-column extraction methods for hexachlorobutadiene in dichloroethylene still bottoms by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

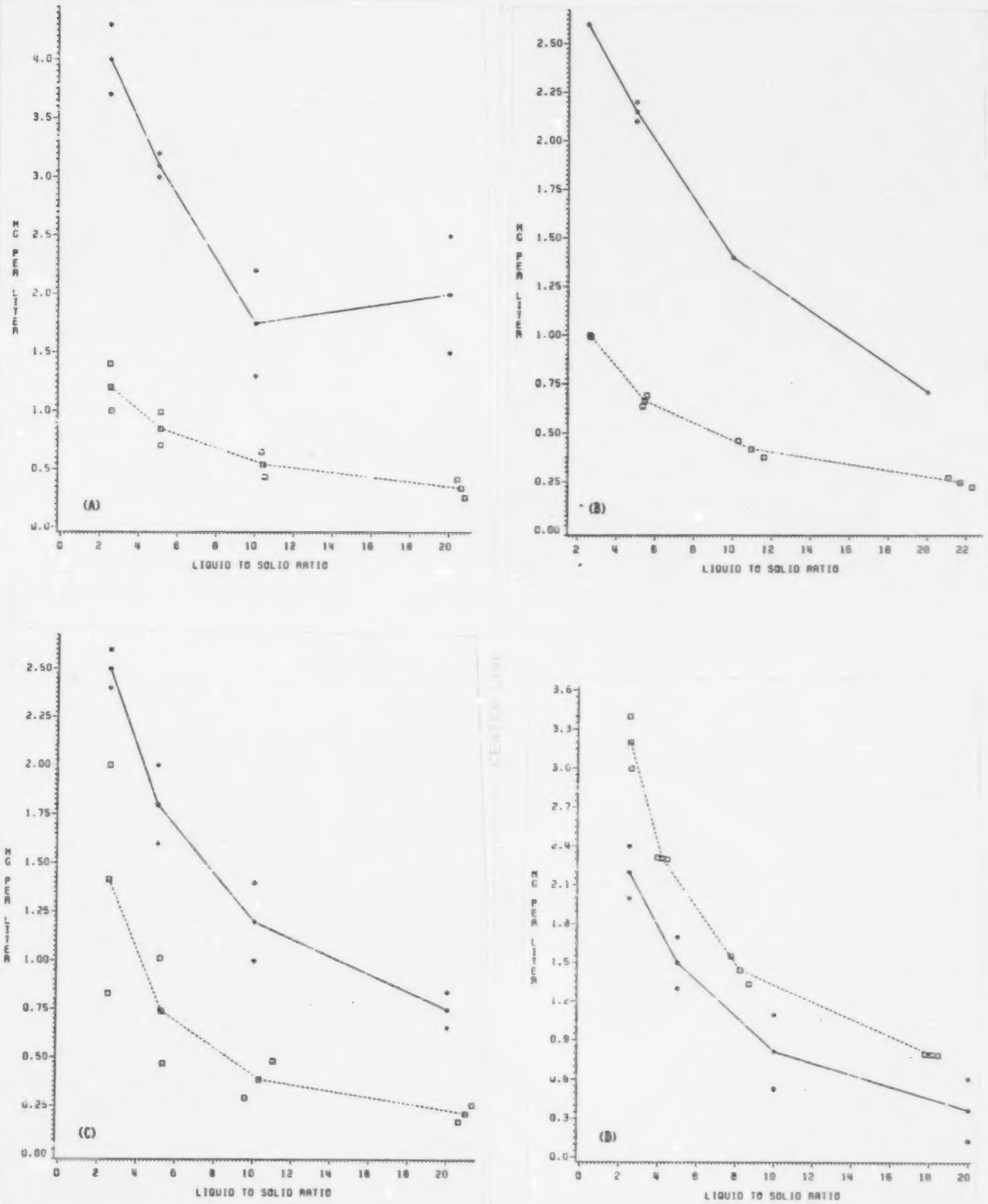


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Fig. 86. Comparison of rotary-batch and upflow-column extraction methods for barium in paint sludge by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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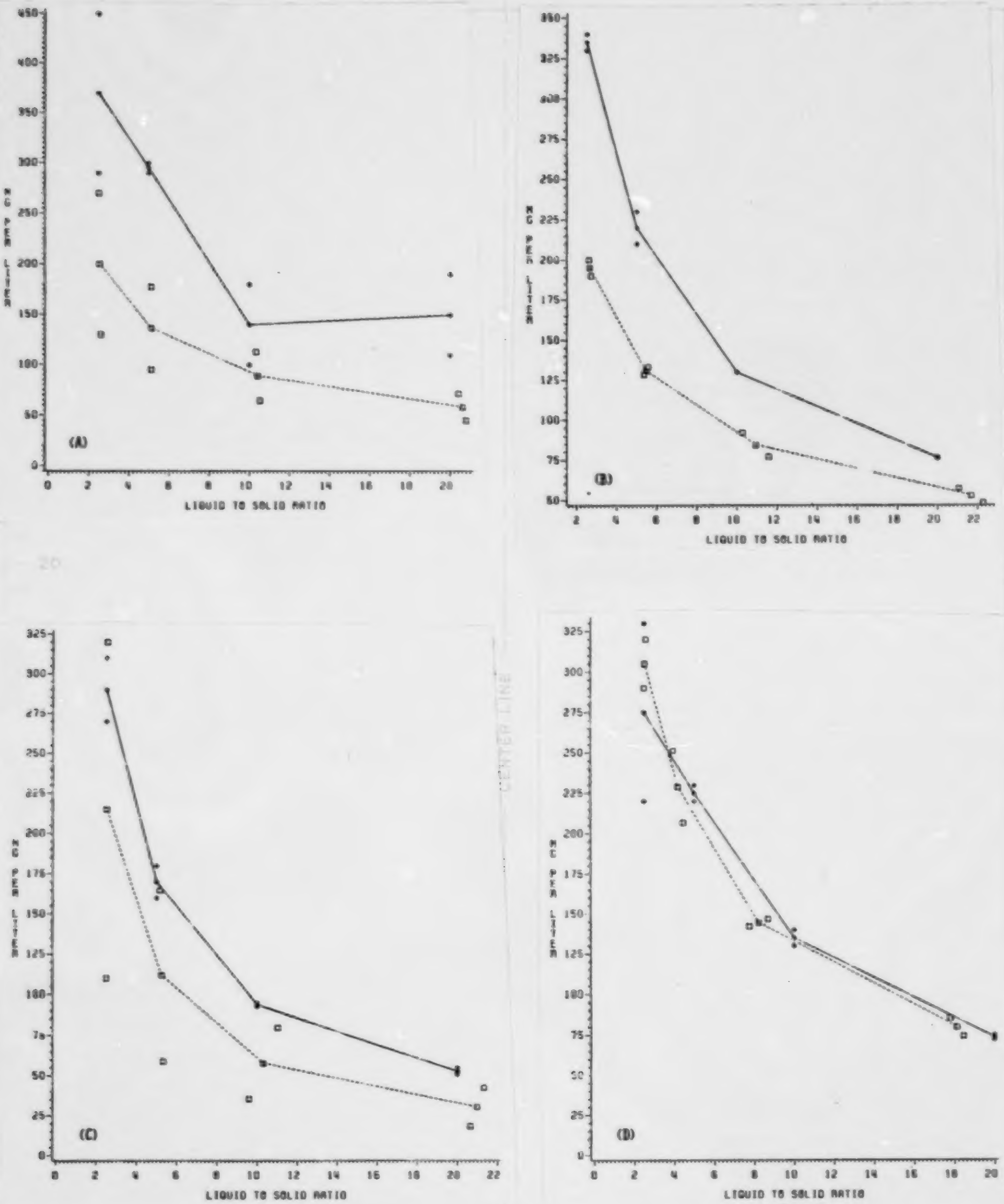

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Fig. 87. Comparison of rotary-batch and upflow-column extraction methods for zinc in paint sludge by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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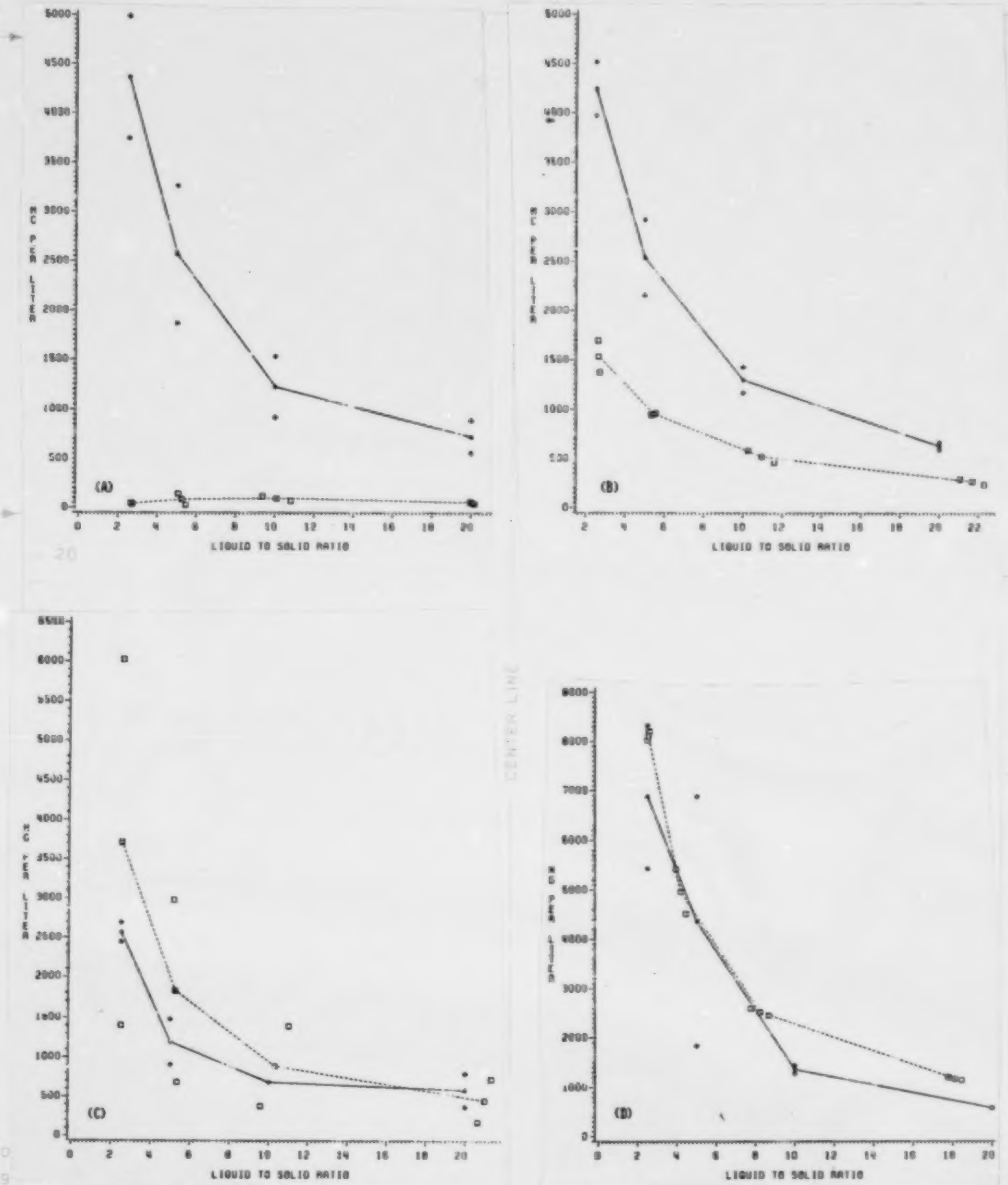

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Fig. 88. Comparison of rotary-batch and upflow-column extraction methods for ethoxyethanol in paint sludge by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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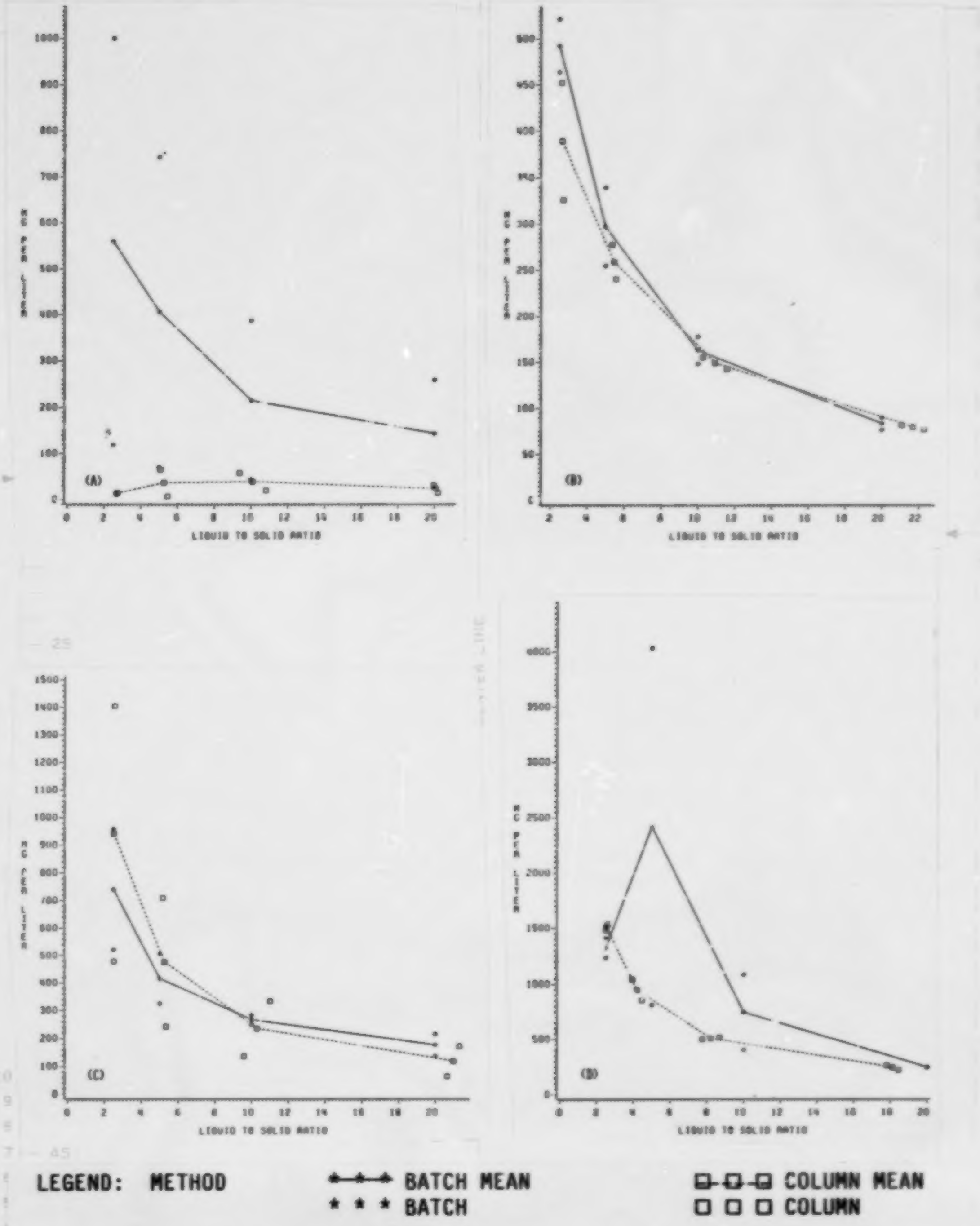
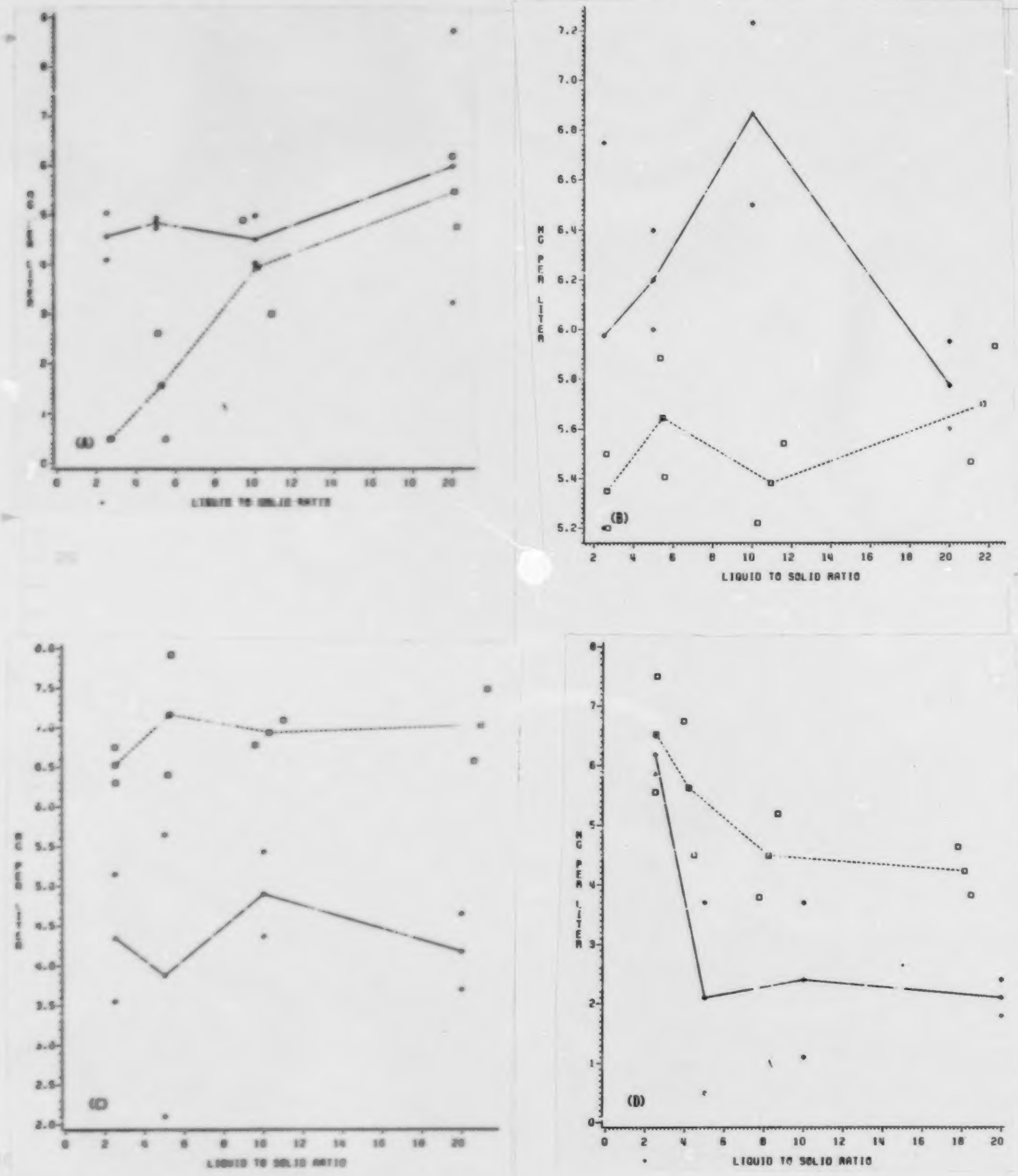


Fig. 89. Comparison of rotary-batch and upflow-column extraction methods for ethoxyethyl acetate in paint sludge by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.



LEGEND: METHOD ●-●-● **BATCH MEAN** ☐-☐-☐ **COLUMN MEAN**
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Fig. 90. Comparison of rotary-batch and upflow-column extraction methods for toluene in paint sludge by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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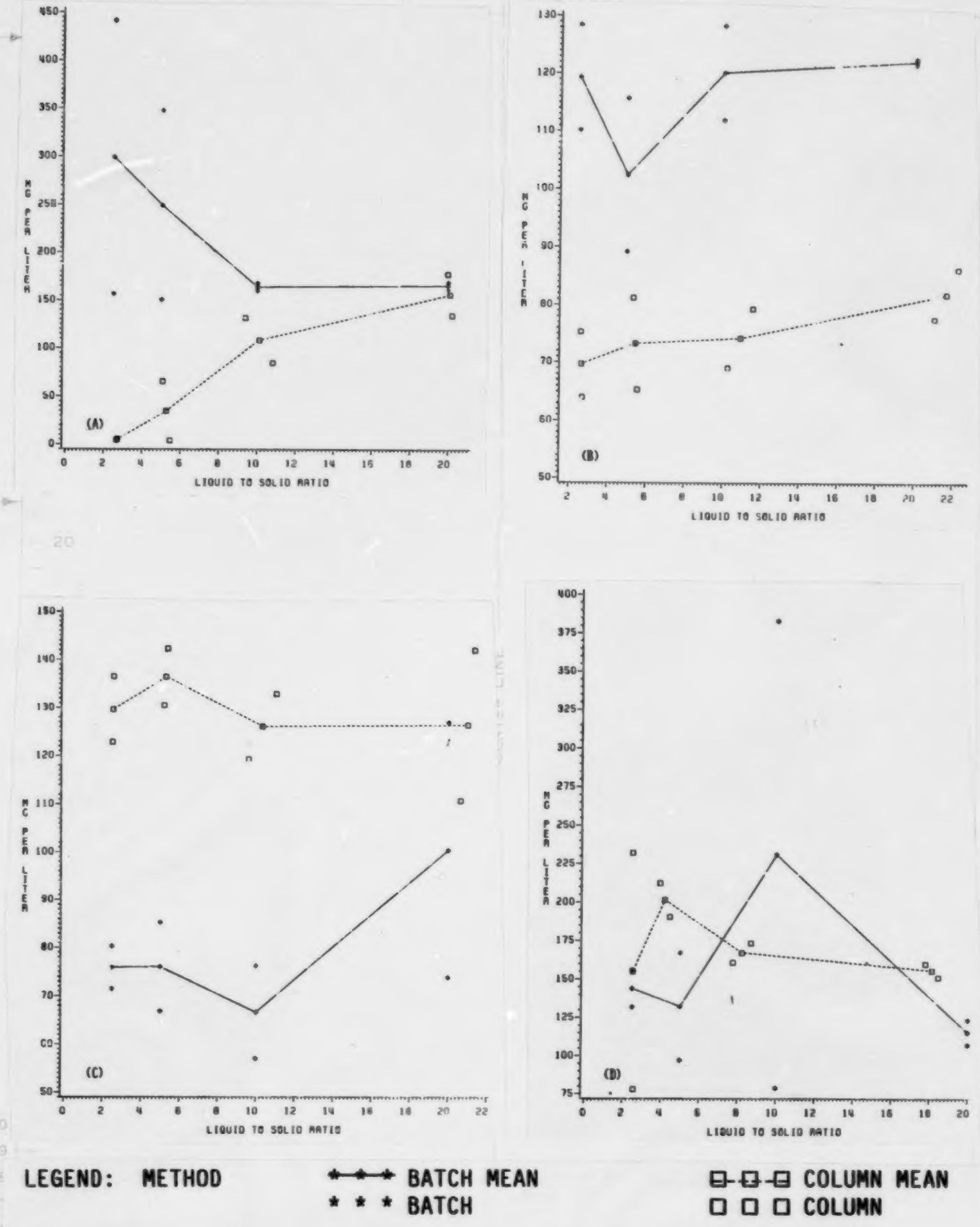

Fig. 91. Comparison of rotary-batch and upflow-column extraction methods for xylenes in paint sludge by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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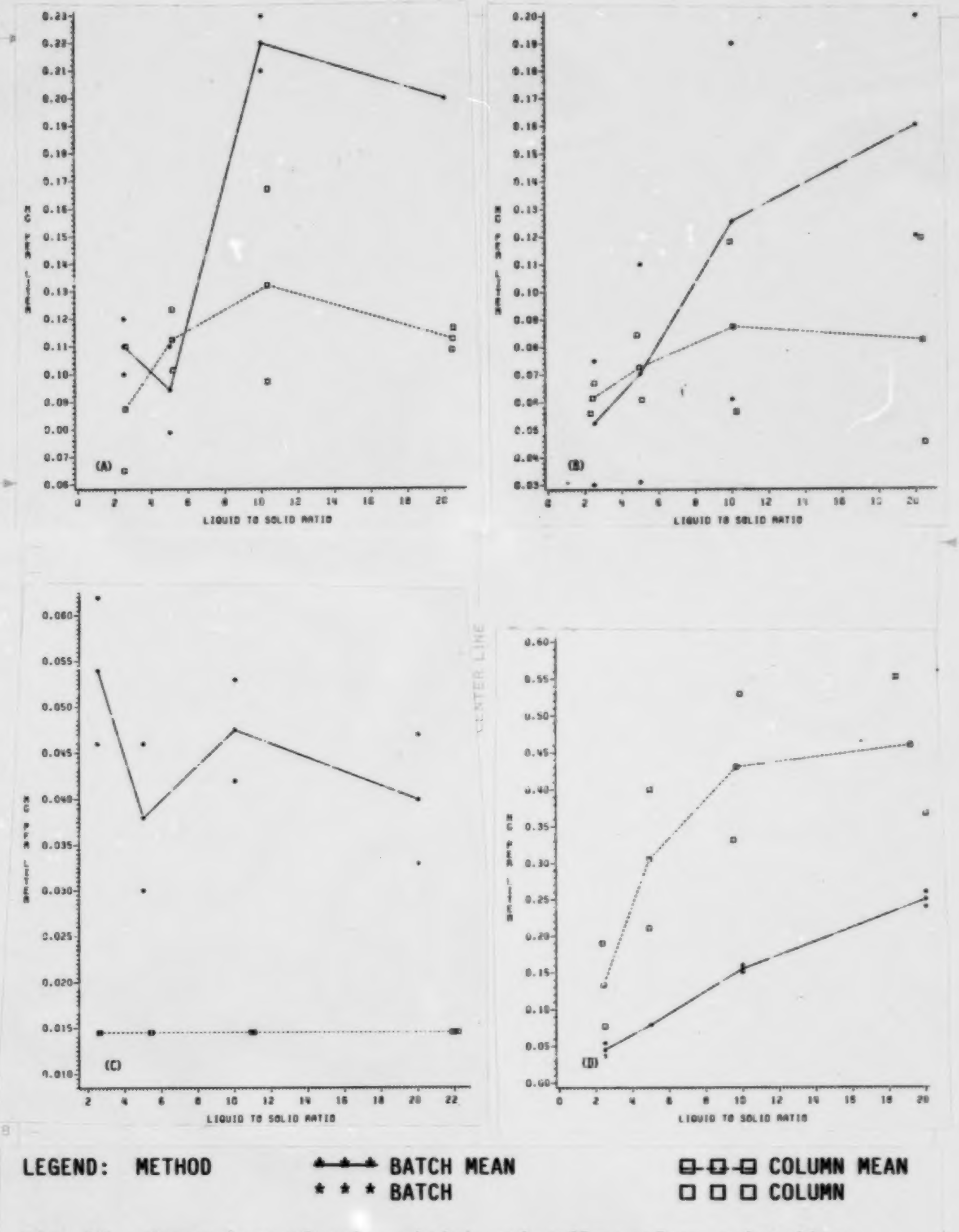

Fig. 92. Comparison of rotary-batch and upflow-column extraction methods for barium in electroplating waste by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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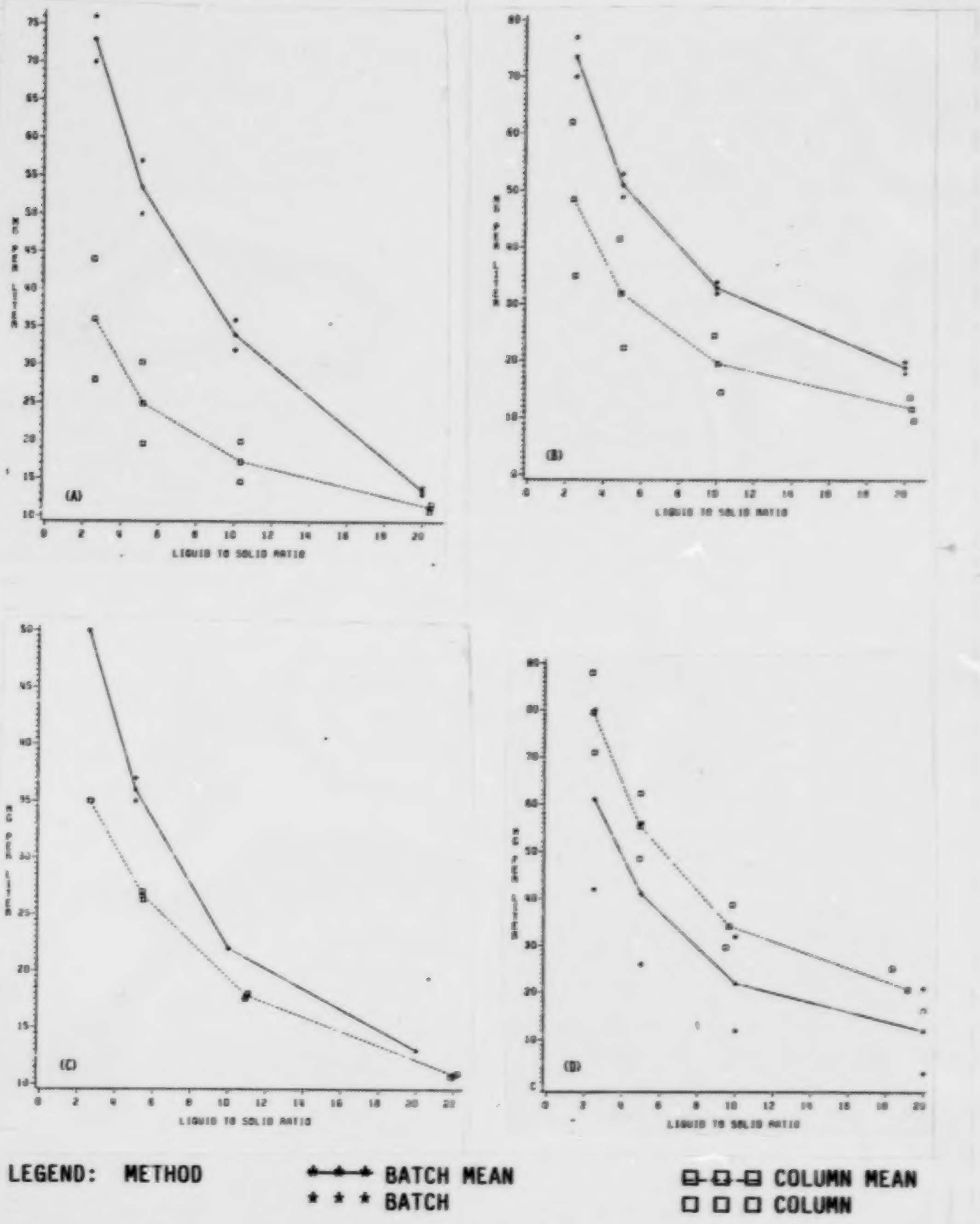

Fig. 93. Comparison of rotary-batch and upflow-column extraction methods for boron in electroplating waste by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

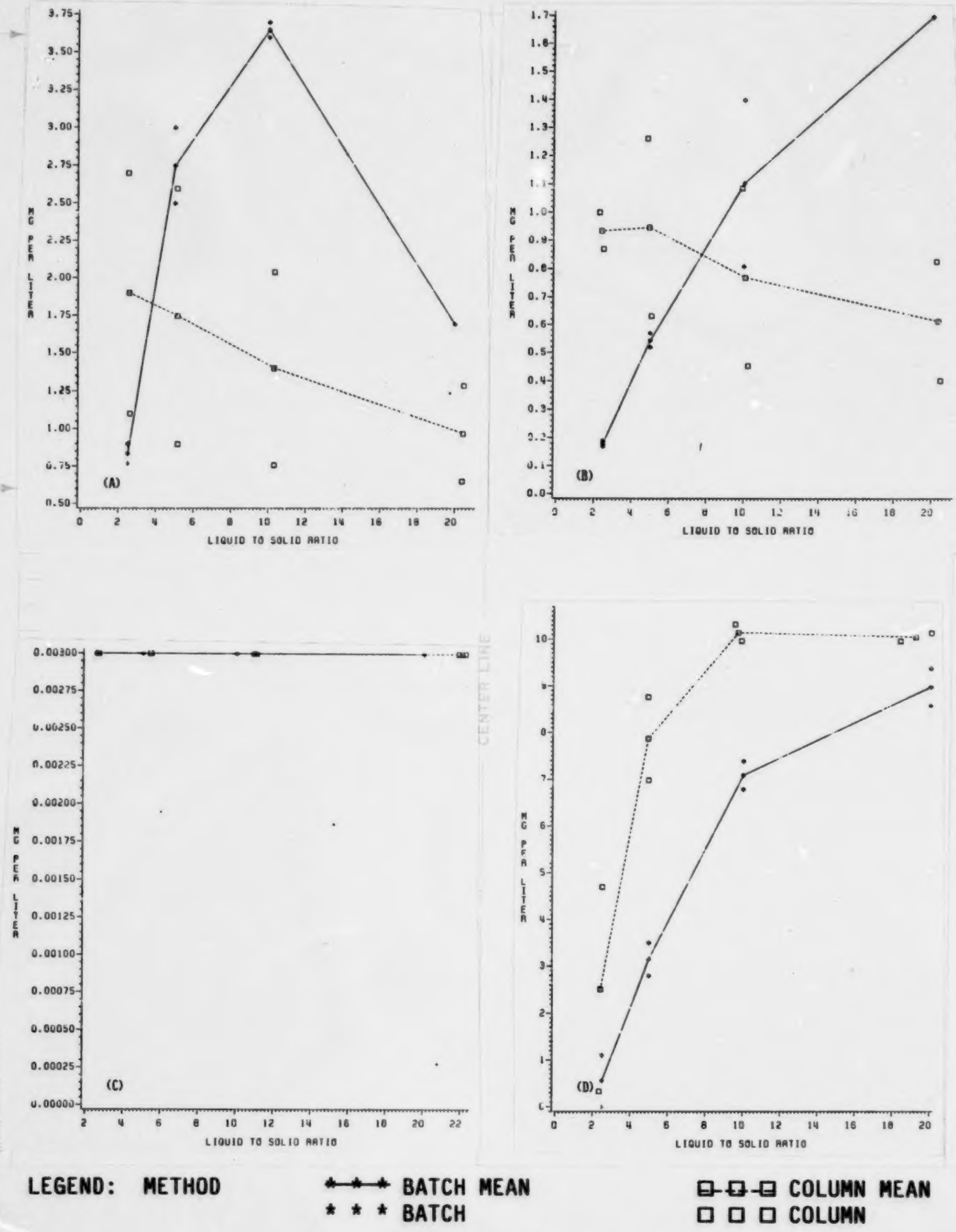

Fig. 94. Comparison of rotary-batch and upflow-column extraction methods for manganese in electroplating waste by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

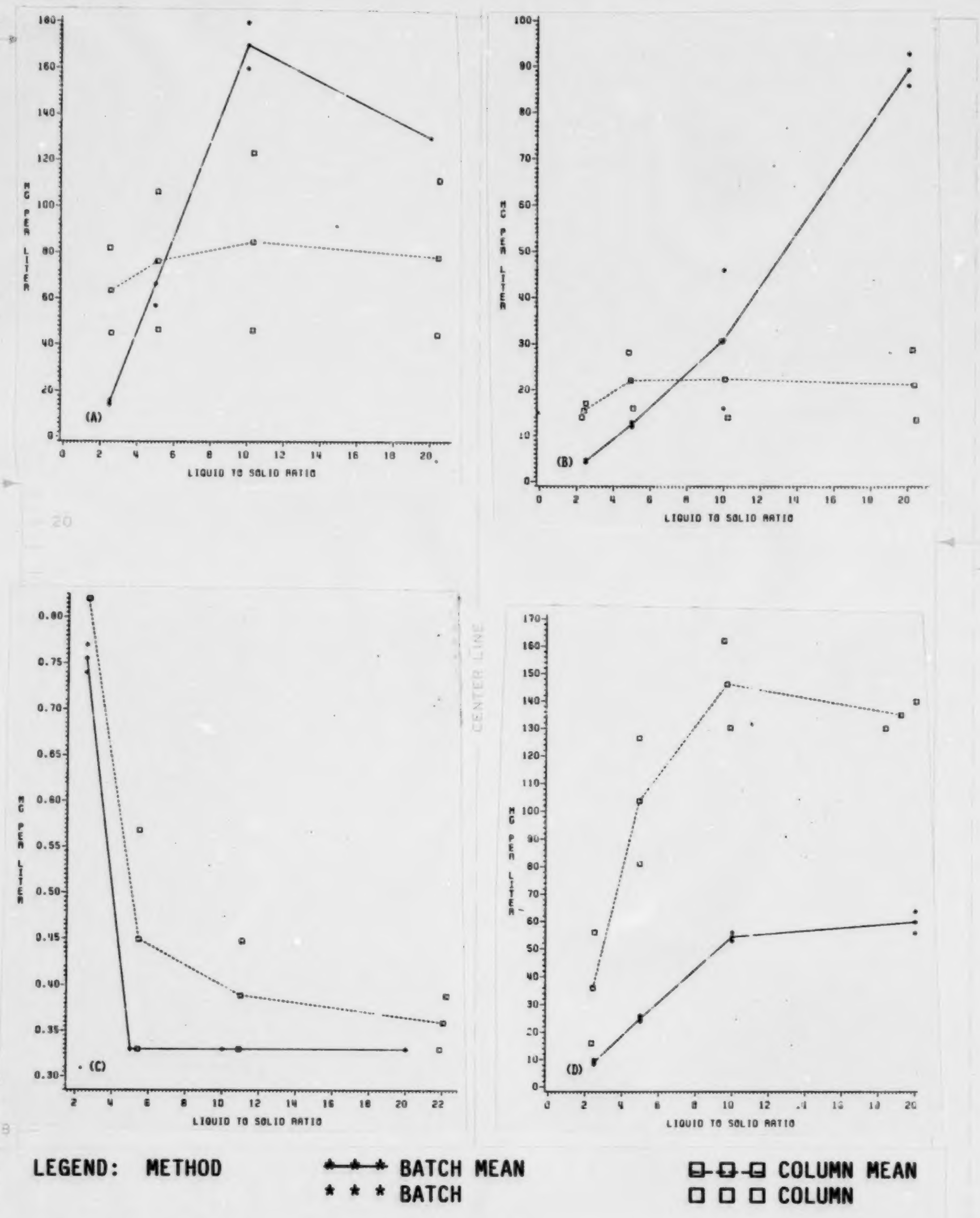

Fig. 95. Comparison of rotary-batch and upflow-column extraction methods for nickel in electroplating waste by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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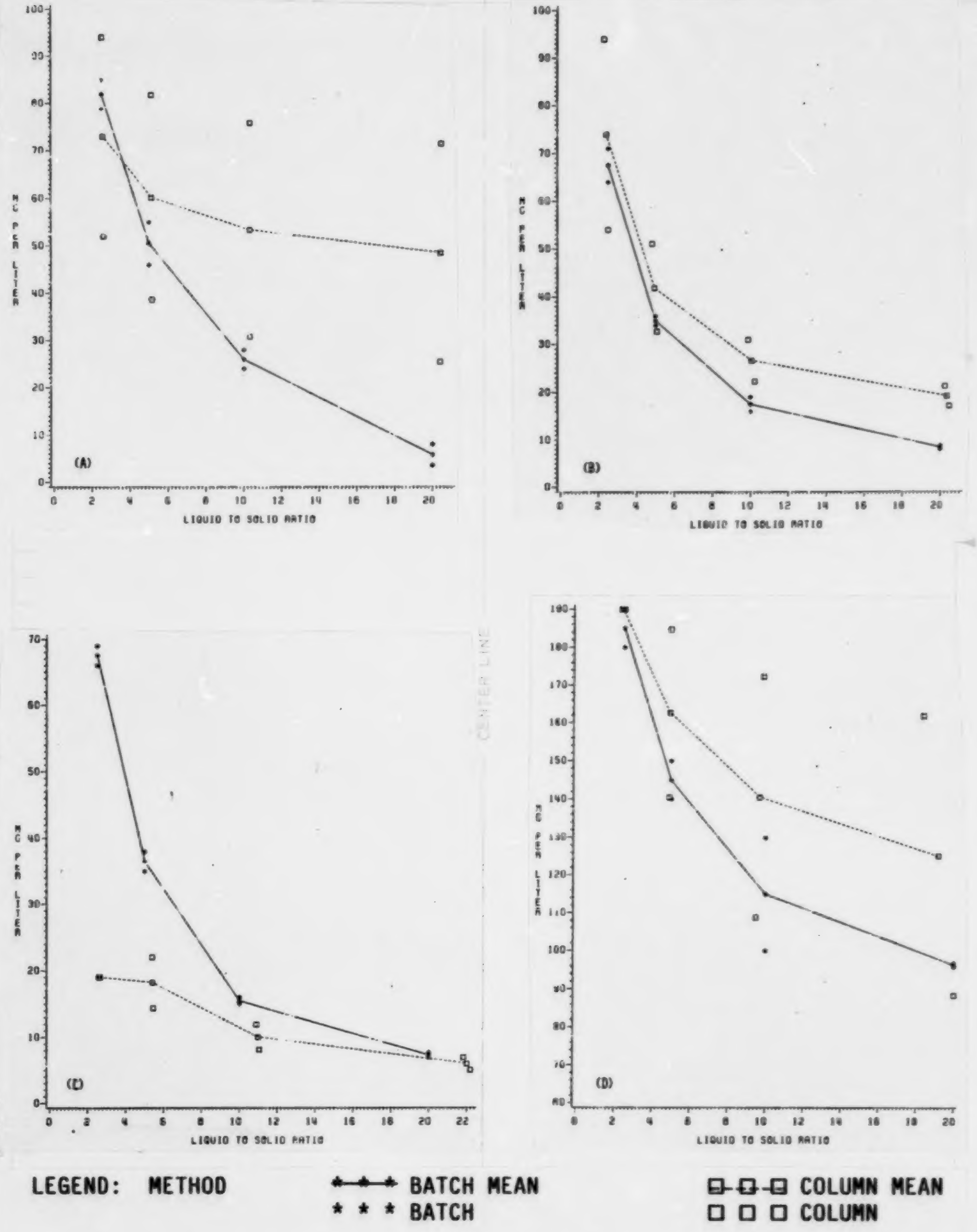

Fig. 96. Comparison of rotary-batch and upflow-column extraction methods for potassium in electroplating waste by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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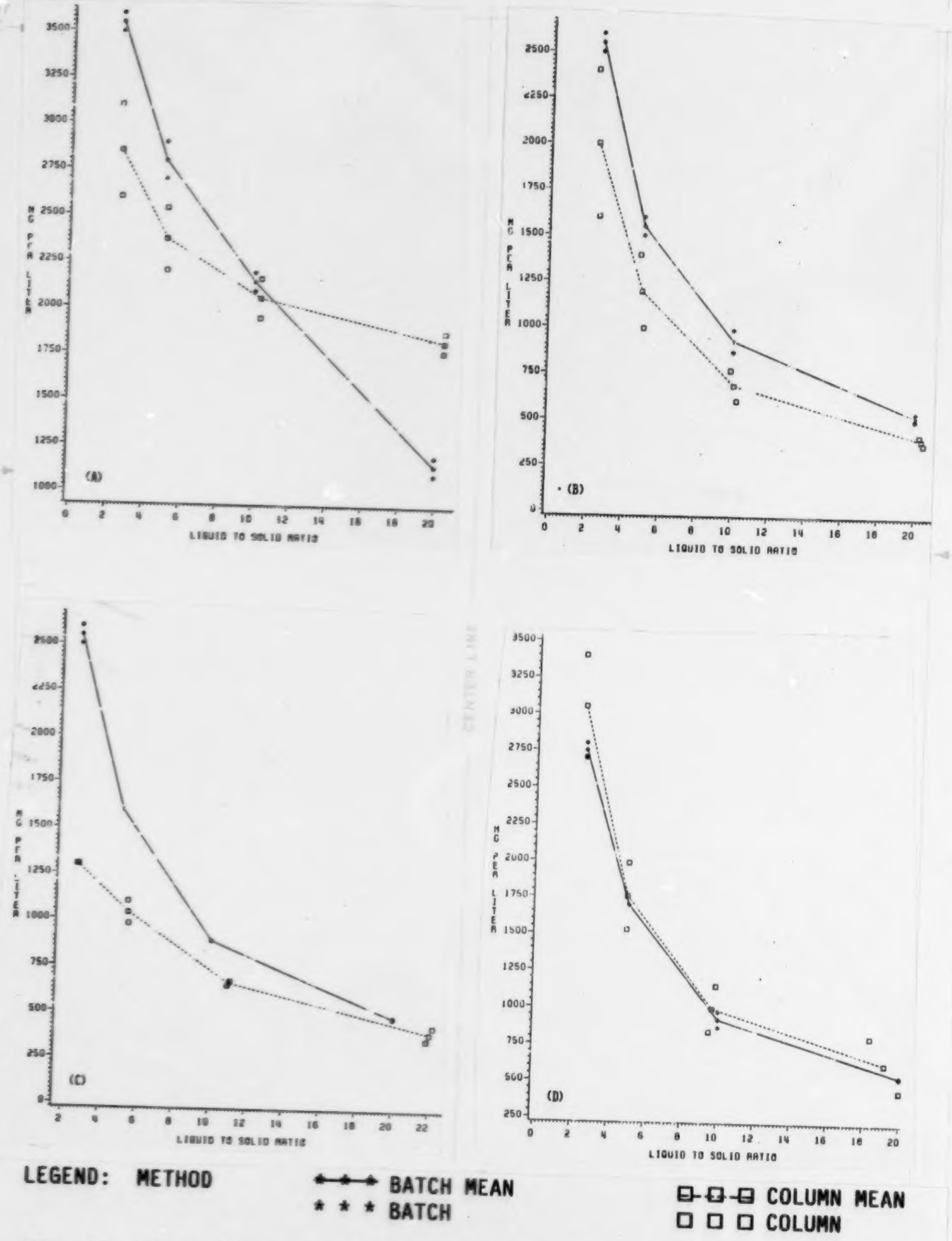

Fig. 97. Comparison of rotary-batch and upflow-column extraction methods for sodium in electroplating waste by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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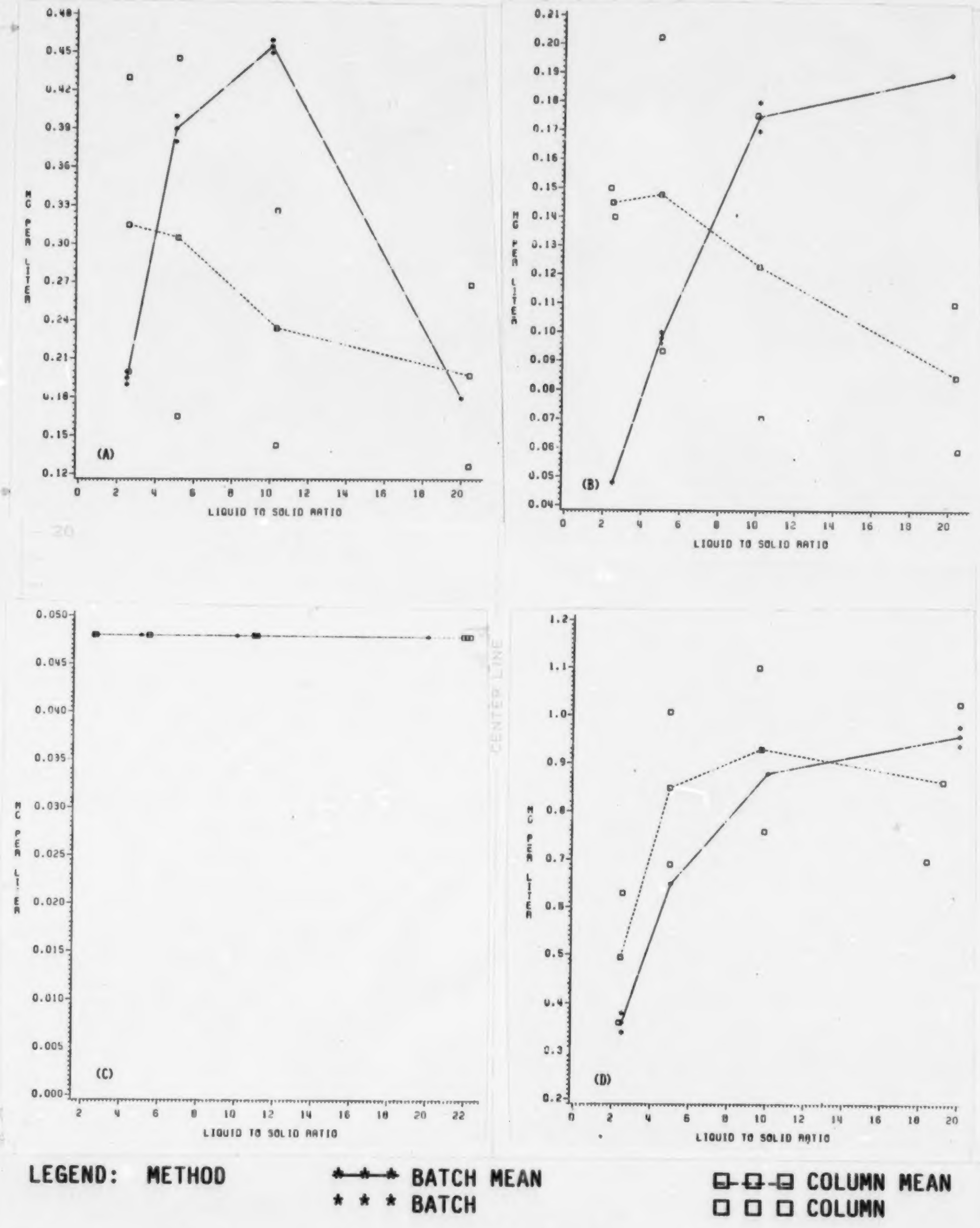

Fig. 98. Comparison of rotary-batch and upflow-column extraction methods for strontium in electroplating waste by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

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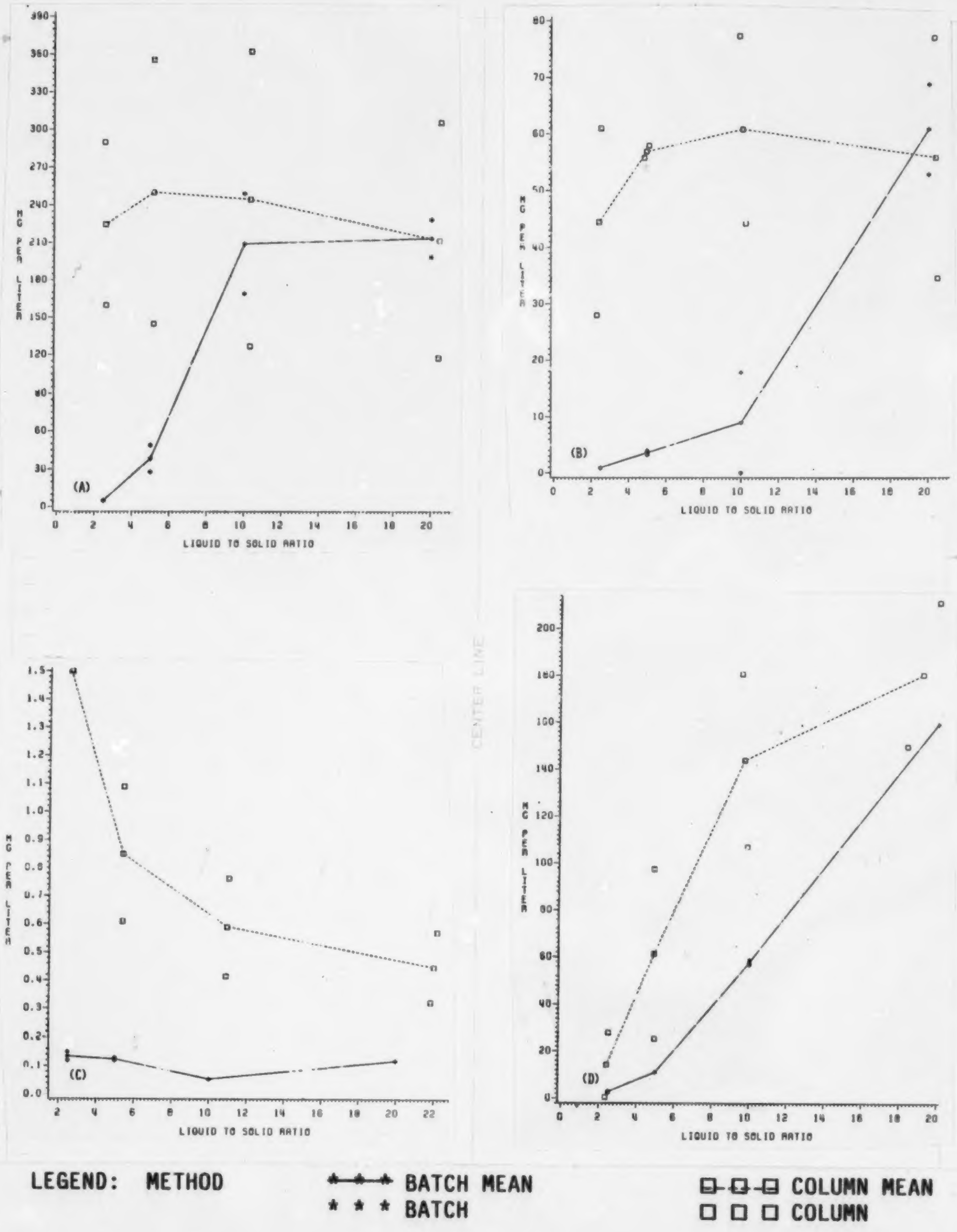

Fig. 99. Comparison of rotary-batch and upflow-column extraction methods for zinc in electroplating waste by four extracting media. (A) acetate buffer, pH 5, (B) carbonic acid, (C) de-ionized water, and (D) ORNL/MWL.

APPENDIX B

TABLE 1. PHYSICOCHEMICAL CHARACTERISTICS OF THE WASTES USED IN THE FIELD AND LABORATORY STUDIES

	API/incinerator* ash	Dichloroethylene* still bottoms	Paint* sludge	Electroplating waste
% Solids	82.8	72.9	42.3	30.3
% Porosity	18.4	33.0	58.8	71.1
		Inorganic elemental concentration (mg/kg)		
Na				
ICP[†]	9.8E3		<0.25E3	1.0E4
NAA[‡]	9.6E3		0.16E3	1.0E4
Mg				
ICP	1.1E4		1.0E4	0.6E3
NAA	1.3E4		1.1E3	0.55E3
K				
ICP	3.4E3		0.49E3	<0.78E3
NAA	2.94E3		0.36E3	
Ca				
ICP	8.9E4		2.0E3	3.0E3
NAA	9.5E4		1.95E3	2.64E3
Cr				
ICP	3.9E3		0.8E3	1.9%
NAA	3.9E3		0.68E3	1.4%
Mn				
ICP	0.41E3		0.037E3	0.19E3
NAA	0.41E3		0.034E3	0.15E3
B (ICP)	<15		70	610
Ba	190		3100	64
Cd	2.9		2.2	<11
Fe	21000		6900	22000
Mo	82		83	630
Ni	40		<22	14000
Pb	1700		1500	590
Sr	260		22	<19
Ti	930		3700	22
V	290		<3	<18
Zn	1800		4100	36000

---

*Wastes as admixtures as described in text.

[†] Analyses determined by Inductive Couple Plasma (ICP).

[‡] Analyses determined by Neutron Activation Analyses (NAA).

TABLE 2. CONCENTRATIONS OF TARGET ORGANIC COMPOUNDS

Waste	Organic compounds	Concentration* (mg/kg)
API/incinerator ash†		
	Toluene	6.1 ± 0.3
	Xylene	1.0 ± 0.2
	Naphthalene	20.5 ± 1.2
Dichloroethylene still bottoms†		
	trans 1,2-dichloroethylene	425 ± 122
	1,2-Dichloroethane	1360 ± 117
	1,1,1-Trichloroethane	630 ± 49
	1,1-Dichloropropane	62.7 ± 2.5
	Trichloroethylene	100 ± 19
	Hexachlorobutadiene	224000 ± 26000
Paint waste†		
	Ethoxyethanol	5290 ± 192
	Toluene	298 ± 51
	Ethoxyethyl acetate	1790 ± 196
	Xylenes	24530 ± 448
	C_3-benzenes	1920 ± 34
Electroplating waste		
	No target organic compounds	

*Average of three analytical replicates.

†Wastes as admixtures as described in text.

TABLE 3. ELEMENTAL CONCENTRATIONS EXTRACTED BY EP EXTRACTIONS*

Element	API/incinerator ash	Dichloroethylene still bottoms	Paint sludge	Electroplating waste	RCRA/EP criteria
		Elements in primary drinking water standard			
As	0.01	<0.0001	0.005	0.002	5
Ba	0.24	0.28	0.60	0.41	100
Cd	<0.0002	<0.0002	0.008	0.009	1
Cr	38	0.0017	0.11	0.31	5
Pb	0.0046	0.0027	0.16	0.037	5
Se	<0.001	0.001	<0.001	<0.001	1
Ag	0.0065	0.0002	<0.0005	<0.0005	5
Hg	0.00006	0.00001	<0.00005	0.0002	0.2
		Major and secondary elements			
K	16	n.a.†	20	70	n.c.‡
Na	240	n.a.	97	910	n.c.
Ca	2200	n.a.	25	72	n.c.
Mg	110	n.a.	1.5	11	n.c.
B	1.8	n.a.	<0.46	16	n.c.
Ni	<0.41	n.a.	0.66	150	n.c.
Zn	<0.15	n.a.	83	430	n.c.

*Concentrations are expressed in mg/L, elements in the Primary Drinking Water Standard were determined by AA methodology and the major secondary elements by ICP methodology.

†n.a. = not analyzed.

‡n.c. = no criteria established for RCRA/EP.

TABLE 4. CONCENTRATIONS OF ORGANIC COMPOUNDS EXTRACTED BY EP EXTRACTIONS*

Waste	Concentration (mg/L)
API/incinerator ash	
Naphthalene	0.069
Dichloroethylene still bottoms	
Dichloroethylene	n.d.
Trichloroethylene	n.d.
Dichloroethane	n.d.
Trichloroethane	n.d.
Hexachlorobutadiene	76.5 ± 0.3
Paint sludge	
Ethoxyethanol	226 ± 29
Ethoxyethyl acetate	125 ± 11
Toluene	2.45 ± 1.11
Xylenes	65.2 ± 8.4

*Average of two extraction replicates and two analytical replicates (N=4), except naphthalene in API/incinerator ash which represents a single replication (2 analytical replicates), n.d. = not detected at 2 mg/L.

TABLE 5. PRECISION OF ICP ANALYSES*

Element	Level (mg/L) ± S.D. (n=10)
Ag	0.99 ± 0.02
Al	1.21 ± 0.03
As	0.68 ± 0.19
B	0.95 ± 0.03
Ba	1.00 ± 0.05
Be	1.02 ± 0.04
Ca	1.02 ± 0.23
Cd	1.00 ± 0.01
Co	0.99 ± 0.02
Cr	1.02 ± 0.04
Cu	1.01 ± 0.03
Fe	1.11 ± 0.06
Ga	1.00 ± 0.01
Hf	1.03 ± 0.05
K (5 mg/L)	5.52 ± 1.19
Li	0.70 ± 0.03
Mg	1.15 ± 0.22
Mn	0.01 ± 0.03
Mo	1.06 ± 0.05
Na (100 mg/L)	97.9 ± 15.2
Ni	1.01 ± 0.03
P	1.02 ± 0.12
Pb	0.97 ± 0.06
Sb	1.17 ± 0.15
Se	0.98 ± 0.08
Si	0.98 ± 0.05
Sn	1.02 ± 0.04
Sr	1.00 ± 0.01
Ti	1.00 ± 0.005
V	1.00 ± 0.00
Zn	1.05 ± 0.07
Zr	1.00 ± 0.00

*At 1 mg/L.

TABLE 6. ANALYSES OF NBS 1633A FLY ASH BY LITHIUM BORATE FUSION AND ICP ANALYSES

Element	Certified value (mg/kg)	No. of analyses	Mean (mg/kg)	Relative standard deviation (%)
Na	1,700	5	2,540	14
K	18,800	7	17,400	16
Ca	11,100	7	10,800	10
Mg	4,550	7	4,350	7
Fe	94,000	7	91,700	4
Al*	140,000	7	145,000	9
Ti*	8,000	7	7,560	5
P†		6	1,950	4
Sr	830 ± 30	6	1,400	9
Ba*	1,500	7	1,400	9
V*	300	7	290	6
Cr	196 ± 6	7	177	5
Mn	190	7	175	5
Co*	46	6	42.3	5
Ni	127 ± 4	7	123	5
Cu	118 ± 3	7	114	7
Mo*	29	7	32.4	11
Pb	72.4 ± 0.4	7	88.8	20
Zn	220 ± 10	7	209	5
Be*	12	7	1206	5
Zr†		7	208	5

*NBS value - not certified.

†No NBS value available.

TABLE 7. EFFICIENCY OF DIETHYLETHER EXTRACTION OF MWL FOR CARBOXYLIC ACIDS

Carboxylic acid	Level (mM)	Recovery (%)
Formic acid	3	50
Acetic acid	3	70
Propionic acid	3	85
Butyric acid	3	90
Pentanoic acid	6	90
Hexanoic acid	6	90
Heptanoic acid	6	90
Octanoic acid	6	90

TABLE 8. OPERATING CONDITIONS FOR MEASUREMENT OF VOLATILE ORGANIC COMPOUNDS IN LEACHATES

Dichloroethylene still bottoms

Instrument:
Perkin-Elmer Model 3920 equipped with Flame Ionization Detector
Column:
3.05 m x 45.7 cm o.d. glass, packed with 60/80 Carbopak B/1% SP1000
Flowrate:
2.1 x 10^5 Pa He
Volume injected:
5 µl
Temperature program:
2 min initial hold at 50°C, 4°C/min to 230°C, and 16-min hold at 230°C
Injection temperature:
270°C
Detection temperature:
300°C
Quantitation:
Single point external standardization at 80 µg/mL, using PE Sigma 10 data system.

Paint sludge

Instrument:
Perkin-Elmer Sigma 1 Analyzer with Flame Ionization Detector
Column:
3.05 m x 45.7 cm o.d. glass, packed with 60/80 Carbopak B/1% SP1000
Flowrate:
3.5 x 10^5 Pa He
Volume injection:
2 µL
Temperature program:
2 min initial hold at 50°C, 4°C/min to 230°C, and 16-min hold at 230°C
Injection temperature:
250°C
Detection temperature:
250°C
Quantitation:
Single point external standardization at 100 µg/mL, using PE Sigma 10 data system.

TABLE 9. PRECISION AND ACCURACY OF ANALYSIS OF VOLATILE ORGANIC COMPOUNDS

Compound	Accuracy/precision	Level (mg/L)	N
Dichloroethylene still bottoms constituents			
Trans-1,2-dichloroethylene	80.7 ± 9.3%	80	20
1,2-Dichloroethane	78.3 ± 15%	80	20
1,1,1-Trichloroethane	78 ± 19.6%	80	20
Trichloroethylene	82.9 ± 22%	80	20
Paint sludge constituents			
Ethoxyethanol	100.8 ± 11.3%	100	40
Ethoxyethyl acetate	108.0 ± 16.6%	100	40
Toluene	100.5 ± 18.8%	100	40
Xylenes	103.8 ± 15.0%	100	40

TABLE 10. RECOVERY OF PAINT SLUDGE CONSTITUENTS FROM LABORATORY MEDIA (%)

	Medium			
Analyte	Distilled water	Acetate	Carbonic acid water	ORNL/MWL
Ethoxyethanol	95	110	106	102
Ethoxyethyl acetate	99	100	102	118
Toluene	100	100	100	106
Xylenes	100	102	101	103

TABLE 11. RECOVERY OF VOLATILE CONSTITUENTS OF DICHLOROETHYLENE STILL BOTTOMS FROM LABORATORY MEDIA (%)

	Medium			
Analyte	Distilled water	Acetate	Carbonic acid water	ORNL/MWL
Trans-1,2-dichloroethylene	102	105	101	108
1,2-Dichloroethane	94	99	99	105
1,1,1-Trichloroethane	102	100	106	109
Trichloroethylene	104	96	98	106

TABLE 12. RECOVERY OF NAPHTHALENE FROM LABORATORY MEDIA

Medium	Spike level (mg/L)	Recovery (%)
Distilled water	0.1	88
Acetate	0.1	84
Carbonic acid	0.1	85
ORNL/MWL	0.1	86

TABLE 13. RECOVERY OF HEXACHLOROBUTADIENE (HCB) FROM EXTRACTION MEDIA*

Medium	Amount added	Amount found
	(mg/L)	
MWL	3.0	3.0 (n=10)
Sodium acetate	13.6	13.6 (n=1)
Carbonic acid	7.2	6.8 (n=1)
Distilled water	7.0	6.6 (n=1)

*No interferences were observed in any of the media.

TABLE 14. CONTENT OF CARBOXYLIC ACIDS IN MWL LEACHATE FROM LYSIMETERS A AND B OVER TIME

Time (days)	Carboxylic acid content (mg/L)																				
	Acetic			Propionic			Butyric			Pentanoic			Hexanoic			Heptanoic			Octanoic		
	Av	Lysimeter		Av	Lysimeter		Av	Lysimeter		Av	Lysimeter		Av	Lysimeter		Av	Lysimeter		Av	Lysimeter	
		A	B		A	B		A	B		A	B		A	B		A	B		A	B
7	1419	1312	1527	607	500	714	1272	1000	1545	673	463	884	649	433	866	80	48	113	134	77	192
14	1236	1140	1333	490	410	571	1243	757	1730	505	421	589	466	400	533	63	61	65	126	115	136
21	1032	882	1191	401	321	516	833	636	1010	420	336	524	383	300	450	44	40	54	96	100	94
27	598	444	752	337	980	1086	965	918	1012	320	332	308	333	341	325	107	100	115	20	20	20
48	1033	980	1086	347	310	385	581	527	636	299	296	303	220	210	230	48	53	43	91	73	109
56	726	687	765	288	287	290	755	854	657	382	215	550	294	355	234	80	101	60	133	169	96
74	1004	829	1179	335	261	409	588	382	794	368	212	525	186	152	221	30	20	40	76	60	93
79	1156	1195	1118	306	381	232	546	763	330	457	641	273	244	308	181	49	78	20	62	104	20
Av	1025			389			848			428			347			63			92		
S.D.	264			108			290			120			152			25			40		

TABLE 15. DISTRIBUTION OF CARBOXYLIC ACIDS IN LEACHATE WITH RESPECT TO TOTAL ORGANIC CARBON*

	Carboxylic acid								
Time (days)	Acetic	Propionic	Butyric	Pentanoic	Hexanoic	Heptanoic	Octanoic	Total carboxylic acid	Total organic carbon (mg/L)
	% of TOC								
7	19.0	9.9	22.9	13.2	13.4	1.7	3.0	83.1	3000
14	18.0	8.7	24.4	10.8	10.5	1.5	3.1	77.0	2750
21	17.6	8.4	19.1	10.5	10.1	1.2	2.7	69.6	2350
48	18.8	7.7	14.3	8.0	6.2	1.4	2.8	59.2	2200
56	14.8	7.2	20.9	11.6	9.3	2.7	4.6	71.1	1950
74	21.2	8.6	16.7	11.4	6.1	1.0	2.7	67.7	1900
79	24.4	7.9	15.5	14.2	8.0	1.7	2.2	73.9	1900
Av.	19.1	8.3	19.1	11.4	9.1	1.6	3.0	71.7	2292
S.D.	3.0	0.8	3.8	2.0	2.6	0.5	0.8	7.5	437

*Average of lysimeters A and B.

TABLE 16. COMPARISON OF CARBOXYLIC ACID CONCENTRATIONS AMONG DIFFERENT SOURCES OF MUNICIPAL WASTE LEACHATE

	Carboxylic acids (mg/L)				
Source of MWL	Acetic	Propionic	Butyric	Pentanoic	Hexanoic
ORNL*	1610±300	830±30	2060±450	880±170	900±140
		(0.51) †	(1.28)	(0.54)	(0.55)
WES-I ‡	1740±860	1170±620	2190±440	1050±360	1620±110
		(0.67)	(1.25)	(0.60)	(0.93)
WES-II ‡	360±50	180±20	390±40	360±60	700±100
		(0.50)	(1.08)	(1.00)	(1.94)

*Samples of leachate (n=2) from lysimeters A and B, July 6, 1982.

†Ratio of the carboxylic acid relative to the concentration of acetic acid of that leachate.

‡Samples of leachate (n=2) taken from lysimeters of approximately 1.5 and 4.7 years of age respectively, WES-I and WES-II (Waterways Experimental Station, Vicksburg, Mississippi).

TABLE 17. CONCENTRATIONS OF INORGANIC CATIONS AND ANIONS IN LYSIMETER MWL OVER TIME

	Time in days*							
	-31	7	14	21	48	56	74	79
				(mg/L)				
Cation								
Calcium	695	450	410	380	370	340	330	310
Iron	nr[†]	60	60	60	60	50	50	60
Magnesium	105	60	50	50	40	40	40	40
Manganese	17	10	10	10	10	10	10	10
Potassium	325	130	120	90	80	60	50	50
Sodium	600	250	230	190	160	140	130	115
Anions								
Sulfate	nr	110	70	<60	<40	<40	<40	<40
Chloride	nr	190	180	180	120	110	90	100

*Time relative to August 6, 1982, when leaching of the industrial wastes was started. Average of both lysimeters (A and B) are rounded to nearest 10 mg/L. Average concentrations were observed to be less than the following for the following consituents: Ag <0.5, Al <0.5, B <4, Ba <0.1, Cd <0.1, Cr <0.2, Cu <0.2, Mo <1, Ni <1, Pb <2, Se <3, V <0.1, Zn <10, NO_3 <40, NO_2 <20, F <0.2, Br <50, and PO_4 <40 mg/L using ICP and anion chromotography methodologies.

[†] Not reported.

TABLE 18. INORGANIC ELEMENTAL CONCENTRATIONS MEASURED IN CONTROL TREATMENTS FROM AGGRESSOR*

Element	Lysimeter A	Lysimeter B
	(mg/L)	
Antimony	0.40 ± 0.97	0.69 ± 0.98
Barium	0.17 ± 0.05	0.22 ± 0.05
Boron	2.63 ± 0.15	2.57 ± 0.05
Cadmium	0.06 ± 0.09	0.08 ± 0.09
Calcium	480 ± 17	557 ± 15
Chromium	0.07 ± 0.16	0.19 ± 0.04
Cobalt	0.14 ± 0.01	0.14 ± 0.06
Iron	56 ± 17	54 ± 21
Magnesium	72 ± 2	75 ± 2
Manganese	11.3 ± 0.6	13.2 ± 0.5
Phosphorus	2.23 ± 3.41	5.57 ± 3.11
Potassium	163 ± 6	170 ± 8
Silicon	23 ± 1	21 ± 1
Sodium	310 ± 10	270 ± 18
Strontium	0.68 ± 0.01	0.68 ± 0.02
Vanadium	0.04 ± 0.10	0.06 ± 0.09
Zinc	15.7 ± 1.5	1.8 ± 0.9

*Concentrations determined by ICP analyses in leachate from sand and sand/sawdust controls (filtered and nonfiltered leachates), n=3 for lysimeter A and n=4 for lysimeter B. Concentrations of the following elements were below detection at the respective concentration (mg/L) Ag (0.09), Al (0.29), As (3.2), Be (0.01), Cu (0.16), Ga (0.35), Hf (0.20), Li (9.8), Mo (0.14), Ni (0.55), Pb (1.3), Se (2.2), Sn (23), Ti (0.02), Zn (0.09).

TABLE 19. INORGANIC ELEMENTAL CONCENTRATIONS MEASURED IN CONTROL TREATMENTS FROM TARGET*

	Lysimeter							
	A				B			
Element	Mean	Std	Min	Max	Mean	Std	Min	Max
	(mg/L)							
Boron	1.57	1.19	0.46[†]	3.80	1.41	1.41	0.46[†]	4.30
Calcium	402.00	126.00	60.00	670.00	456.00	116.00	240.00	650.00
Iron	28.10	17.90	0.12[†]	78.00	50.90	27.10	0.10[†]	110.00
Magnesium	66.40	17.50	35.00	100.00	60.80	18.10	33.00	89.00
Manganese	9.74	2.93	1.30	15.00	11.10	2.37	4.20	15.00
Potassium	157.00	100.00	34.00	350.00	197.00	129.00	47.00	580.00
Sodium	216.00	107.00	87.00	440.00	216.00	83.00	95.00	400.00
Zinc[‡]	4.48	6.04	0.11[†]	21.00	0.54	0.96	0.11[†]	4.60

*Concentrations in leachate from sand and sand/sawdust controls (filtered and nonfiltered leachates) over 79 d, n=58 for lysimeter A and n=59 for lysimeter B.

[†] Detection level by ICP.

[‡] Mean levels of zinc were significantly different ($P<0.05$) between lysimeter A and B.

TABLE 20. COMPARISON OF LEACHATE CHARACTERISTICS

	Study number*						
Element	1	2	3	4	5	6	7
pH	3.5-6.0	5.3-6.5	4.3-5.5	5.9-7.0	2.8-7.0	5.1-7.2	5.7-7.6
				(mg/L)			
TOC	1380-3200	n.a.[†]	7000-12000	30-1700	120-10000	70-27700	n.a.
Calcium	260-500	50-4080	650-1120	99-400	90-1750	76-3900	200-2100
Iron	22-79	100-860	230-360	11-230	9-1380	0.5-2200	0.06-1500
Potassium	37-140	500-3770	360-615	20-220	40-2050	35-2300	31-560
Magnesium	37-61	100-420	130-180	13-96	13-450	35-1140	120-780
Sodium	99-260	100-1440	710-940	35-460	55-750	44-1580	33-1240
Sulfate	40-129	50-1000	n.a.	10-100	n.a.	7-1560	n.d.[‡]-1800
Chloride	60-212	100-2300	870-1440	n.a.	90-3900	60-2470	2-5590
Nitrate	<40	n.a.	n.a.	45-1020	n.a.	2-46	n.d.-1125

*Study number references

1 - ORNL (this study).
2 - Qasim and Burchinal 1976 (Lysimeter study).
3 - Meyers et al. 1980 (Lysimeter study).
4 - Johansen and Carlson 1976 (Six natural leachates).
5 - Fuller et al. 1979 (three natural leachates).
6 - Chian and DeWalle 1977 (twelve natural leachates).
7 - Kmet and McGinley 1982 (sixteen natural leachates).

[†] n.a. = not analyzed.

[‡] n.d. = not dectected.

TABLE 21. COMPARISON OF CARBOXYLIC ACID CONCENTRATIONS IN LEACHATE

	Study number*		
	1	2	3
		(mg/L)	
Acetic	680-1530	10-2750	510-6690
Propionic	230-1100	10-4370	255-3180
Butyric	330-1730	10-5870	510-9630
Pentanoic	210-900	10-550	305-2180
Hexanoic	150-870	90-600	565-2400
Heptanoic	20-110	n.a.†	n.a.
Octanoic	20-190	n.a.	n.a.

*Study number-references
1 - ORNL (this study).
2 - Johansen and Carlson 1976 (Table 3) (eight natural leachates).
3 - Chian and DeWalle 1977 (Table 16) (four natural leachates).

†n.a. = not analyzed.

TABLE 22. LIQUID/SOLID RATIOS AND pH OF FINAL LEACHATE FROM AGGRESSOR*

	Number of columns	Liquid/solid ratio	pH
API/incinerator ash	4	19.8 ± 2.6	8.27 ± 0.28
Dichloroethylene			
still bottoms	4	17.9 ± 6.5	5.35 ± 0.03
Paint sludge	4	19.1 ± 4.3	5.41 ± 0.02
Electroplating waste	4	21.1 ± 1.8	6.28 ± 0.15
Controls			
sand	2	16.7 ± 4.6	5.77 ± 0.55
Sand/sawdust	2	20.8 ± 2.1	5.30 ± 0.44

*28 days leaching, liquid/solid ratios calculated by dividing the volume of leachate collected (mL) by the weight of the waste (800 g).

TABLE 23. INORGANIC ELEMENTAL CONCENTRATIONS (ICP) MEASURED IN LEACHATES FROM AGGRESSOR*

	Waste				Controls	
Element	API/incinerator ash	Dichloroethylene still bottoms	Paint sludge	Electroplating waste	Sand	Sand/Sawdust
			(mg/L)			
Barium	0.17 ± 0.01	0.18 ± 0.06	0.44 ± 0.08	0.46 ± 0.3	0.17 ± 0.05	0.23 ± 0.04
Boron	2.91 ± 0.14	2.71 ± 0.28	3.11 ± 0.23	24 ± 1	2.62 ± 0.13	2.57 ± 0.06
Calcium	1200 ± 53	545 ± 70	557 ± 66	560 ± 41	520 ± 44	530 ± 53
Chromium	12.7 ± 6.6	0.17 ± 0.02	0.15 ± 0.03	0.14 ± 0.00†	0.09 ± 0.15	0.20 ± 0.04
Iron	2.69 ± 3.24	34.5 ± 23.1	47.7 ± 11.7	15.2 ± 16.4	60.0 ± 17.1	48.0 ± 19.9
Magnesium	89 ± 16	76 ± 7	76 ± 7	86 ± 2	73 ± 2	74 ± 3
Manganese	1.54 ± 0.55	13.2 ± 16	12.9 ± 1.5	13.9 ± 0.8	12.2 ± 1.0	12.7 ± 1.5
Molybdenum	0.39 ± 0.06	0.02 ± 0.23	0.08 ± 0.19	0.25 ± 0.06	0.02 ± 0.00 †	0.06 ± 0.35
Nickel	0.66 ± 0.0†	0.55 ± 0.00†	0.55 ± 0.00†	123 ± 11	0.55 ± 0.00†	0.55 ± 0.00†
Phosphorus	2.0 ± 0.0†	3.74 ± 2.64	4.76 ± 1.67	5.91 ± 0.88	3.20 ± 4.20	5.40 ± 2.27
Potassium	257 ± 8	243 ± 127	286 ± 119	202 ± 8	165 ± 6	170 ± 10
Sodium	837 ± 38	312 ± 46	323 ± 28	1008 ± 38	282 ± 33	293 ± 15
Strontium	2.10 ± 0.20	0.70 ± 0.05	0.68 ± 0.04	1.07 ± 0.05	0.68 ± 0.01	0.68 ± 0.03
Zinc	0.38 ± 0.52	9.73 ± 7.39	71.4 ± 21.1	175 ± 20	8.85 ± 8.86	6.23 ± 6.74

*An underlined concentration indicates that the concentration is significantly different ($P<0.05$) than the concentration of that element in the leachates of the control treatment using Duncan's multiple range test. Concentrations are average values (and standard deriations) calculated from four replicate leachates, filtered and nonfiltered (n=8), except for controls which represent two replicate treatments, filtered and nonfiltered (n=4).

†ICP detection limit for that particular matrix.

TABLE 24. CONCENTRATIONS OF TARGET INORGANIC ELEMENTS MEASURED IN AGGRESSOR LEACHATES

Waste	Element	Concentration*
		(mg/L)
API/incinerator ash		
	Arsenic	0.001 ± 0.000
	Cadmium	0.001 ± 0.000
	Chromium	6.050 ± 2.121
	Lead	0.001 ± 0.000
Paint sludge		
	Barium	0.445 ± 0.056
	Chromium	0.129 ± 0.013
	Lead	0.016 ± 0.005
Electroplating waste		
	Chromium	0.057 ± 0.0005
	Lead	0.001 ± 0.000
	Nickel	118.750 ± 6.142
Controls		
Sand	Arsenic	0.002 ± 0.000
	Cadmium	0.001 ± 0.000
	Chromium	0.101 ± 0.011
	Lead	0.003 ± 0.000
	Nickel	0.220 ± 0.010
Sand/sawdust		
	Arsenic	0.003 ± 0.002
	Cadmium	0.005 ± 0.003
	Chromium	0.097 ± 0.003
	Lead	0.004 ± 0.003
	Nickel	0.240 ± 0.020

*Determined by AAS on four filtered replicate leachates, except for the controls which represent only two replicates, rounded to 0.001 mg/L.

TABLE 25. INORGANIC ELEMENTAL CONCENTRATIONS IN FILTERED AND NONFILTERED API/INCINERATOR ASH LEACHATES (AGGRESSOR)*

Element	Filtered	Nonfiltered
	(mg/L)	
Barium	0.16 ± 0.01	0.18 ± 0.01
Boron	2.90 ± 0.18	2.92 ± 0.10
Calcium	1200 ± 82	1200 ± 0
Chromium	7.42 ± 4.75	18.0 ± 2.4
Cobalt	0.04 ± 0.08†	0.08 ± 0.00
Iron	0.12 ± 0.00†	5.5 ± 1.9
Magnesium	89 ± 18	89 ± 17
Manganese	1.31 ± 0.44	1.77 ± 0.61
Molybdenum	0.42 ± 0.07	0.35 ± 0.05
Potassium	262 ± 5	252 ± 9
Sodium	840 ± 29	835 ± 51
Strontium	2.10 ± 0.18	2.10 ± 0.24
Vanadium	0.09 ± 0.00†	0.15 ± 0.02
Zinc	0.10 ± 0.25	0.67 ± 0.60

*Average concentrations and standard deviations calculated from ICP analyses of four replicated leachates.

†At least one of the four ICP analyses was at the detection limit for that solution matrix.

TABLE 26. INORGANIC ELEMENTAL CONCENTRATIONS IN FILTERED AND NONFILTERED DICHLOROETHYLENE STILL BOTTOMS LEACHATES (AGGRESSOR)*

Element	Filtered	Nonfiltered
	(mg/L)	
Aluminum	0.14 ± 0.50	0.88 ± 0.32
Antimony	0.17 ± 1.03	0.13 ± 0.96
Barium	0.16 ± 0.07	0.19 ± 0.05
Boron	2.85 ± 0.35	2.57 ± 0.10
Cadmium	0.09 ± 0.02	0.09 ± 0.04
Calcium	575 ± 91	515 ± 29
Chromium	0.16 ± 0.01	0.17 ± 0.03
Cobalt	0.15 ± 0.06	0.14 ± 0.06
Iron	18.0 ± 20.9	51.0 ± 9.1
Magnesium	80.2 ± 8.8	72.7 ± 0.5
Manganese	13.7 ± 2.1	12.7 ± 1.0
Molybdenum	0.02 ± 0.25[†]	0.05 ± 0.23
Phosphorus	2.45 ± 2.89	5.02 ± 1.88
Potassium	275 ± 164	212 ± 92
Silicon	25 ± 3	22 ± 2
Sodium	335 ± 39	290 ± 45
Strontium	0.72 ± 0.08	0.69 ± 0.01
Titanium	0.01 ± 0.03	0.01 ± 0.03[†]
Vanadium	0.01 ± 0.09	0.01 ± 0.10
Zinc	9.51 ± 8.37	9.95 ± 7.58

*Average concentrations and standard deviations calculated from ICP analyses of four replicated leachates.

[†] At least one of the four ICP analyses was at the detection limit for that solution matrix.

TABLE 27. INORGANIC ELEMENTAL CONCENTRATIONS IN FILTERED AND NONFILTERED PAINT SLUDGE LEACHATES (AGGRESSOR)*

Element	Filtered	Nonfiltered
	(mg/L)	
Antimony	0.12 ± 0.95	0.51 ± 0.82
Barium	0.42 ± 0.08	0.46 ± 0.08
Boron	3.27 ± 0.22	2.95 ± 0.06
Cadmium	0.09 ± 0.02	0.07 ± 0.09
Calcium	587 ± 79	527 ± 38
Chromium	0.15 ± 0.13	0.16 ± 0.04
Cobalt	0.57 ± 0.17	0.54 ± 0.08
Iron	43 ± 15	52 ± 6
Magnesium	79 ± 8	72 ± 3
Manganese	13.5 ± 1.9	12.2 ± 1.0
Molybdenum	0.05 ± 0.23	0.11 ± 0.17
Phosphorus	3.85 ± 1.26	5.67 ± 1.65
Potassium	325 ± 151	247 ± 79
Silicon	25 ± 1	23 ± 2
Sodium	337 ± 93	310 ± 34
Strontium	0.71 ± 0.05	0.66 ± 0.02
Vanadium	0.07 ± 0.00†	0.01 ± 0.10
Zinc	75 ± 24	67 ± 21

*Average concentrations and standard deviations calculated from ICP analyses of four replicated leachates.

†At least one of the four ICP analyses was at the detection limit for that solution matrix.

TABLE 28. INORGANIC ELEMENTAL CONCENTRATIONS IN FILTERED AND NONFILTERED ELECTROPLATING WASTE LEACHATES (AGGRESSOR)*

Element	Filtered	Nonfiltered
	(mg/L)	
Barium	0.45 ± 0.03	0.46 ± 0.02
Boron	24 ± 1	24 ± 1
Calcium	572 ± 49	547 ± 33
Cobalt	0.38 ± 0.03	0.37 ± 0.05
Iron	0.58 ± 1.08	29.7 ± 7.7
Magnesium	88 ± 3	86 ± 1
Manganese	14.2 ± 1.0	13.5 ± 0.58
Molybdenum	0.25 ± 0.05	0.03 ± 0.6
Nickel	125 ± 13	122 ± 9
Phosphorus	5.5 ± 0.9	6.3 ± 0.7
Potassium	207 ± 9	197 ± 5
Silicon	5.6 ± 0.8	7.0 ± 0.7
Sodium	1025 ± 50	992 ± 15
Strontium	1.07 ± 0.05	1.07 ± 0.05
Zinc	175 ± 17	175 ± 25

*Average concentrations and standard deviations calculated from ICP analyses of four replicated leachates.

TABLE 29. FRACTION OF THE INORGANIC ELEMENTS LEACHED FROM THE INDUSTRIAL WASTES BY MWL (AGGRESSOR)[*]

Waste	Element	Fraction leached (%)
API/incinerator ash		
	Calcium	14.6
	Chromium	6.4
	Potassium	57.1
	Magnesium	2.6
	Molybdenum	8.9
	Sodium	112
	Strontium	10.8
Paint sludge		
	Barium	0.13
	Zinc	30.3
Electroplating waste		
	Barium	9.5
	Boron	73.9
	Potassium	79.9
	Manganese	1.7
	Molybdenum	0.9
	Sodium	153
	Nickel	18.5
	Strontium	<43
	Zinc	9.7

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[*] For those elements in which the concentration in the industrial waste leachate was significantly higher than that measured in the control leachate (Table 23), except P in the electroplating waste where fraction could not be calculated as total concentration of P in waste was not determined.

TABLE 30. CONCENTRATIONS OF TARGET ORGANIC COMPOUNDS MEASURED IN LEACHATES OF AGGRESSOR

Waste	Organic compounds	Concentration* (mg/L)
API/incinerator ash		
	Naphthalene	0.12 ± 0.03
Dichloroethylene still bottoms		
	Dichloroethane	40.6 ± 26.6
	Trichloroethane	18.2 ± 37.1
	Trichloroethylene	6.64 ± 6.1
	Hexachlorobutadiene	4.41 ± 1.56
Paint sludge		
	Ethoxyethanol	339 ± 173
	Ethoxyethyl acetate	146 ± 37.9
	Toluene	17.7 ± 10.4
	Xylenes	177 ± 79

*Average and standard deriation of four replicate leachates.

TABLE 31. FRACTIONS OF ORGANIC COMPOUNDS LEACHED FROM THE INDUSTRIAL WASTES BY MWL (AGGRESSOR)

Waste	Compounds	% Leached
API/incinerator ash		
	Naphthalene	11.5
Dichloroethylene still bottoms		
	Dichloroethane	58
	Trichloroethane	56
	Trichloroethylene	128
	Hexachlorobutadiene	0.04
Paint sludge		
	Ethoxyethanol	123
	Ethoxyethyl acetate	161
	Toluene	130
	Xylenes	14

TABLE 32. COMPARISON OF EP EXTRACTS AND AGGRESSOR LEACHATES FOR THE API/INCINERATOR ASH*

Constituents	EP (mg/L)	Aggressor (mg/L)	EP/aggressor
Inorganic			
Calcium	2200	680	3.2
Chromium	38	6.0	6.3
Magnesium	110	16	6.8
Molybdenum	0.30	0.37	0.81
Potassium	16	92	0.17
Sodium	240	555	0.43
Strontium	3.8	1.42	2.68
Organic			
Naphthalene	0.069	0.12	0.58
pH	6.4	8.3	

*Concentrations for EP extracts reflect a single extraction, while concentrations for Aggressor leachates represent the average from four replicates after subtraction of the average concentrations measured in the control leachates.

TABLE 33. COMPARISON OF EP EXTRACTS AND AGGRESSOR LEACHATES FOR THE DICHLOROETHYLENE STILL BOTTOMS*

Constituents	EP (mg/L)	Aggressor (mg/L)	EP/aggressor
Organic			
Dichloroethylene	n.d.†	20.9	--
Dichloropropane	n.d.	<1	--
Trichloroethylene	n.d.	6.63	--
Dichloroethane	n.d.	40.6	--
Trichloroethane	n.d.	18.2	--
Hexachlorobutadiene	76.5	4.41	17
pH	3.50	5.35	--

*Concentrations for EP extracts represent an average of two extractions while concentrations for Aggressor leachates are averages after subtraction of that measured in the control leachates.

†n.d. = not detected.

TABLE 34. COMPARISON OF EP EXTRACTS AND AGGRESSOR LEACHATES FOR THE PAINT SLUDGE*

Constituents	EP (mg/L)	Aggressor (mg/L)	EP/aggressor
Inorganic			
Barium	0.60	0.21	2.9
Zinc	83	65	1.3
Organic			
Ethoxyethanol	226	339	0.67
Toluene	2.4	18	0.13
Ethoxyethyl acetate	125	146	0.86
Xylenes	65	177	0.36
pH	5.0	5.4	

*Concentrations for EP extracts reflect a single extraction for inorganic elements and an average of two extractions for organic compounds. Concentrations for Aggressor leachates represent an average of four leachates after subtraction of the average concentrations measured in the control leachates.

TABLE 35. COMPARISON OF EP EXTRACTS AND AGGRESSOR LEACHATES FOR THE ELECTROPLATING WASTE*

Constituents	EP (mg/L)	Aggressor (mg/L)	EP/aggressor
Inorganic			
Barium	0.41	0.29	1.41
Boron	16	21	0.76
Chromium	0.31	0.14	2.21
Manganese	3.0	1.2	2.5
Molybdenum	0.35	0.22	1.59
Nickel	150	123	1.22
Phosphorus	9.9	2.7	3.6
Potassium	70	37	1.89
Sodium	910	726	1.25
Strontium	n.d.†	0.39	--
Zinc	430	166	2.59
pH	5.1	6.3	

*Concentrations for EP extracts reflect a single extraction, while concentrations for Aggressor leachates represent the average from four replicates after subtraction of the average concentrations measured in the control leachates.

†n.d = not detected by ICP.

TABLE 36. LIQUID/SOLID RATIOS FROM TARGET*

Waste	Number of columns	Liquid/solid ratio		
		Average	Lower †	Upper †
API/incinerator ash	4	17.7	17.5	17.9
Dichloroethylene still bottoms	4	20.8	19.0	22.6
Paint sludge	4	18.0	16.7	19.3
Electroplating waste	4	17.5	16.8	18.2
Average		18.5	17.0	20.0

*After 79 d leaching, calculated by dividing cumulative volume of leachate collected by weight of waste in column.

†Represents the lower and upper 95% confidence levels.

TABLE 37. MLC TARGET CONCENTRATIONS*

Waste	Chemical	Concentrations (mg/L)			Average liquid/solid ratio
		Average	Lower †	Upper †	
API/incinerator ash					
	Calcium	1188	1095	1280	5.4
	Chromium	63	25	101	2.6
	Molybdenum	2.2	0.82	3.6	3.9
	Potassium	562	121	1002	1.5
	Sodium	1904	797	3010	1.5
	Strontium	3.9	1.9	6.0	2.0
	Naphthalene	0.92	0.45	1.4	3.7
Dichloroethylene still bottoms					
	Dichloroethane	49	40	58	8.6
	Trichloroethane	83	67	98	1.4
	Trichloroethylene	97	67	162	5.2
	Hexachlorobutadiene	2651	0	7631	0.9
Paint sludge					
	Barium	2.3	2.0	2.6	0.82
	Zinc	220	165	274	1.6
	Ethoxyethanol	4729	3679	5778	0.72
	Ethoxyethyl acetate	1892	1307	2476	0.72
	Toluene	39	23	56	2.2
	Xylenes	614	286	942	1.5
Electroplating waste					
	Barium	0.47	0.34	0.60	6.3
	Boron	148	139	156	0.69
	Manganese	7.4	5.9	8.9	5.6
	Nickel	147	126	168	7.6
	Potassium	125	0	300	2.3
	Sodium	7058	6577	7540	0.69
	Strontium	1.1	0.98	1.3	4.1
	Zinc	149	107	191	14.0

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*The MLC target concentration is defined as the average at the maximum concentrations in the four replicated leachates, after the subtraction of the corresponding control leachate concentration of that replicate.

†Represents the lower and upper 95% confidence levels.

TABLE 38. AMC8 TARGET CONCENTRATIONS*

Waste	Chemical	Concentrations (mg/L)		
		Average	Lower †	Upper †
API/incinerator ash				
	Calcium	787	668	906
	Chromium	22	11	32
	Molybdenum	0.6	0.32	0.89
	Potassium	164	69	260
	Sodium	617	444	791
	Strontium	2.1	1.4	2.7
	Naphthalene	0.21	0.16	0.26
Dichloroethylene still bottoms				
	Dichloroethane	26	22	31
	Trichloroethane	53	39	67
	Trichloroethylene	26	17	31
	Hexachlorobutadiene	90	0	221
Paint sludge				
	Barium	0.35	0.20	0.52
	Zinc	77	54	100
	Ethoxyethanol	1055	868	1241
	Ethoxyethyl acetate	405	347	463
	Toluene	17	13	20
	Xylenes	269	201	337
Electroplating waste				
	Barium	0.28	0.19	0.38
	Boron	51	46	56
	Manganese	1.3	0	3.3
	Nickel	107	93	121
	Potassium	30	0	88
	Sodium	1209	1059	1358
	Strontium	0.32	0.23	0.41
	Zinc	85	60	110

*The AMC8 target concentration is defined as the average concentration (from four replicated leachates after the subtraction of control concentrations) of a leaching interval over an 8:1 liquid/solid ratio centered around the MLC liquid/solid ratio. If the MLC liquid/solid ratio is less than 4 then AMC8 is average integrated concentration over the first 8:1 liquid/solid ratio.

†Represents the lower and upper 95% confidence levels.

TABLE 39. AMC20 TARGET CONCENTRATIONS[*]

		Concentrations (mg/L)		
Waste	Chemical	Average	Lower[†]	Upper[†]
API/incinerator ash				
	Calcium	771	680	861
	Chromium	11	0.5	16
	Molybdenum	0.30	0.13	0.46
	Potassium	76	38	114
	Sodium	258	188	328
	Strontium	1.5	1.1	1.9
	Naphthalene	0.18	0.15	0.21
Dichloroethylene still bottoms				
	Dichloroethane	26	23	30
	Trichloroethane	43	33	54
	Trichloroethylene	21	19	22
	Hexachlorobutadiene	45	0	97
Paint sludge				
	Barium	0.14	0.10	0.20
	Zinc	35	32	38
	Ethoxyethanol	430	354	506
	Ethoxyethyl acetate	165	142	188
	Toluene	9.9	7.8	12
	Xylenes	174	145	204
Electroplating waste				
	Barium	0.29	0.20	0.38
	Boron	22	19	24
	Manganese	0.79	0	2.5
	Nickel	93	80	106
	Potassium	39	7.5	71
	Sodium	472	385	559
	Strontium	0.34	0.25	0.43
	Zinc	85	60	110

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[*] The AMC20 target concentration is defined as the average concentration (from four replicated leachates after the subtraction of control concentrations) of a leaching interval over an 20:1 liquid/solid ratio centered on th MLC liquid/solid ratio. If the MLC liquid/solid ratio is less that 10 then AMC20 is the average integrated concentration over the first 20:1 liquid/solid ratio.

[†] Represents the lower and upper 95% confidence levels.

TABLE 40. AMC40 TARGET CONCENTRATIONS*

Waste	Chemical	Concentrations (mg/L) Average	Lower †	Upper †
API/incinerator ash				
	Calcium	774	691	857
	Chromium	6.2	2.7	9.6
	Molybdenum	0.09	0	0.27
	Potassium	40	24	57
	Sodium	105	70	141
	Strontium	1.3	0.7	1.9
	Naphthalene	0.17	0.15	0.19
Dichloroethylene still bottoms				
	Dichloroethane	25	17	32
	Trichloroethane	38	24	51
	Trichloroethylene	26	17	34
	Hexachlorobutadiene	31	4.8	56
Paint sludge				
	Barium	0.07	0.0	0.10
	Zinc	20	18	22
	Ethoxyethanol	219	180	257
	Ethoxyethyl acetate	84	72	95
	Toluene	6.9	3.7	10
	Xylenes	136	87	185
Electroplating waste				
	Barium	0.30	0.21	0.40
	Boron	11	9.3	13
	Manganese	1.5	0	3.3
	Nickel	99	85	112
	Potassium	41	0	84
	Sodium	209	150	268
	Strontium	0.36	0.27	0.45
	Zinc	79	57	101

*The AMC40 target concentration is defined as the average concentration (from four replicated leachates after the subtraction of control concentrations) of a leaching interval over a 40:1 liquid/solid ratio centered on the MLC liquid/solid ratio. If the MLC liquid/solid ratio is less than 20 then AMC40 is the average integrated concentration over the first 40:1 liquid/solid ratio.

†Represents the lower and upper 95% confidence levels.

TABLE 41. IAC8 TARGET CONCENTRATIONS*

Waste	Chemical	Concentrations (mg/L) Average	Lower †	Upper †
API/incinerator ash				
	Calcium	792	684	900
	Chromium	21	11	32
	Molybdenum	0.66	0.39	0.93
	Potassium	164	69	260
	Sodium	617	444	791
	Strontium	2.1	1.4	2.7
	Naphthalene	0.21	0.15	0.27
Dichloroethylene still bottoms				
	Dichloroethane	30	28	32
	Trichloroethane	53	40	67
	Trichloroethylene	22	11	34
	Hexachlorobutadiene	90	0	221
Paint sludge				
	Barium	0.35	0.20	0.52
	Zinc	72	59	85
	Ethoxyethanol	1055	868	1241
	Ethoxyethyl acetate	405	347	463
	Toluene	1	13	20
	Xylenes	269	201	337
Electroplating waste				
	Barium	0.25	0.16	0.34
	Boron	51	46	56
	Manganese	0	0	0.15
	Nickel	80	67	92
	Potassium	29	0	88
	Sodium	1209	1059	1359
	Strontium	0.31	0.22	0.39
	Zinc	49	37	62

*The IAC8 target concentration is defined as the average integrated concentration (from four replicated leachates after the subtraction of control concentrations) over the first 8:1 liquid/solid ratio.

†Represents the lower and upper 95% confidence levels.

TABLE 42. FRACTION OF THE INORGANIC ELEMENTS EXTRACTED FROM THE INDUSTRIAL WASTES BY MWL (TARGET)

Waste	Element	Fraction extracted
		%
API/incinerator ash	Calcium	13.4
	Chromium	18.8
	Potassium	41.3
	Molybdenum	7.7
	Sodium	54
	Strontium	10.7
Paint sludge	Barium	0.1
	Zinc	16.3
Electroplating waste	Barium	7.3
	Boron	70.4
	Manganese	2.2
	Nickel	11.3
	Potassium	>59
	Sodium	97
	Strontium	1.8
	Zinc	74

TABLE 43. FRACTION OF ORGANIC COMPOUNDS LEACHED FROM INDUSTRIAL WASTES BY MWL (TARGET)*

Waste	Compounds	% Leached
API/incinerator ash		
	Naphthalene	14.1
Dichloroethylene still bottoms		
	Dichloroethane	37.2
	Trichloroethane	133
	Trichloroethylene	431
	Hexachlorobutadiene	0.4
Paint sludge		
	Ethoxyethanol	161
	Ethoxyethyl acetate	182
	Toluene	63.0
	Xylenes	13.2

*Cumulative quantity of each compound measured in leachates collected over 79 d divided by the quantity measured in the waste (X100).

TABLE 44. THE INFLUENCE OF PROCEDURE, MEDIA, AND LIQUID/SOLID RATIOS ON EXTRACTION OF TARGET CONSTITUENTS

	Variables*									
	Procedure		Media				Liquid/solid ratio			
Waste/Target constituent	Batch	Column	Acetate	Carbonic acid	Water	ORNL/MWL	2.5:1	5:1	10:1	20:1
API/incinerator ash										
Calcium	678^{b}	793^{a}	814^{a}	557^{d}	643^{b}	929^{a}	672^{a}	746^{a}	777^{a}	749^{a}
Chromium	60.5^{a}	57.2^{a}	60.2^{ab}	68.6^{a}	51.3^{b}	55.4^{b}	106^{a}	64^{b}	39^{c}	25^{d}
Molybdenum	0.65^{a}	0.54^{b}	0.64^{a}	0.60^{ab}	0.50^{b}	0.66^{a}	1.0^{a}	0.65^{b}	0.43^{c}	0.30^{d}
Potassium	340^{a}	184^{b}	191^{b}	156^{b}	179^{b}	521^{a}	468^{a}	274^{b}	151^{c}	124^{d}
Sodium	1362^{a}	1016^{b}	2102^{a}	702^{c}	732^{c}	1220^{b}	1906^{a}	1280^{b}	898^{c}	682^{d}
Strontium	6.09^{a}	5.74^{a}	6.46^{a}	3.79^{b}	6.14^{a}	7.27^{a}	8.94^{a}	6.63^{b}	4.74^{c}	3.34^{d}
Naphthalene	0.086^{a}	0.044^{b}	0.050^{bc}	0.118^{a}	0.058^{b}	0.034^{c}	0.064^{ab}	0.043^{b}	0.071^{a}	0.081^{a}
Dichloroethylene Still Bottoms										
Dichloroethane	5.13^{a}	1.59^{a}	0.89^{b}	7.43^{a}	1.07^{b}	4.06^{ab}	5.16^{a}	3.55^{a}	3.18^{a}	1.56^{a}
Trichloroethane	4.29^{a}	2.01^{a}	2.92^{b}	0.00^{b}	1.12^{b}	8.56^{a}	4.38^{a}	3.31^{a}	2.59^{a}	2.40^{a}
Trichloroethylene	28.3^{a}	14.2^{a}	2.3^{b}	0.28^{b}	5.1^{b}	77.0^{a}	9.8^{a}	23.4^{a}	20.5^{a}	30.9^{a}
Hexachlorobutadiene	583^{a}	37.9^{b}	762^{a}	67.1^{b}	204^{b}	210^{b}	867^{a}	350^{b}	33^{b}	26^{b}
Paint sludge										
Barium	1.8^{a}	0.98^{b}	1.7^{a}	1.1^{b}	1.1^{b}	1.5^{a}	2.3^{a}	1.6^{b}	0.9^{c}	0.68^{d}
Zinc	189^{a}	132^{b}	179^{a}	153^{ab}	127^{b}	183^{a}	273^{a}	190^{b}	109^{c}	72^{d}
Ethoxyethanol	2239^{a}	1700^{b}	1146^{b}	1500^{b}	1490^{b}	3742^{a}	3932^{a}	2311^{b}	1075^{c}	559^{c}
Ethoxyethyl acetate	544^{a}	373^{a}	180^{b}	239^{b}	421^{b}	995^{a}	747^{a}	658^{a}	291^{b}	140^{b}
Toluene	5.06^{a}	4.66^{a}	3.78^{b}	5.86^{a}	5.62^{a}	4.17^{b}	4.93^{a}	4.54^{a}	4.91	5.05^{a}
Xylenes	143^{a}	113^{a}	148^{ab}	95.4^{c}	105^{bc}	162^{a}	125^{a}	126^{a}	132^{a}	128^{a}
Electroplating waste										
Barium	0.10^{b}	0.14^{a}	0.13^{b}	0.09^{c}	0.03^{d}	0.23^{a}	0.07^{b}	0.10^{b}	0.15^{a}	0.16^{a}
Boron	37.9^{a}	29.9^{b}	32.9^{b}	36.0^{ab}	25.8^{c}	40.7^{a}	58.5^{a}	40.0^{b}	25.0^{c}	13.9^{d}
Manganese	2.02^{a}	2.49^{a}	1.87^{b}	0.84^{c}	$<0.01^{d}$	6.3^{a}	0.86^{c}	2.13^{b}	3.02^{a}	3.01^{a}
Nickel	41.8^{a}	50.4^{a}	85.5^{a}	27.3^{b}	0.45^{c}	71.2^{a}	19.3^{c}	38.4^{b}	63.7^{a}	64.3^{a}
Potassium	60.1^{a}	66.7^{a}	49.9^{b}	36.3^{c}	22.3^{d}	145^{a}	94.7^{a}	68.6^{b}	50.5^{c}	39.6^{c}
Sodium	1666^{a}	1457^{b}	2345^{a}	1230^{c}	1125^{c}	1544^{b}	2575^{a}	1757^{b}	1166^{c}	748^{d}
Strontium	0.29^{a}	0.30^{a}	0.28^{b}	0.13^{c}	0.05^{d}	0.75^{a}	0.21^{b}	0.31^{a}	0.36^{a}	0.32^{a}
Zinc	48.5^{b}	100^{a}	175^{a}	36.6^{c}	0.41^{c}	79.1^{b}	39.1^{c}	52.9^{bc}	91.0^{ab}	111^{a}

*Target constituents having the same letter superscript within procedure, media, or liquid/solid ratio are not significantly different ($P<0.05$) using Duncan's multiple range test.

TABLE 45. AVERAGE pH OF INDUSTRIAL WASTE LABORATORY EXTRACTS*

Waste	Media	Liquid/solid ratios							
		2.5		5.0		10		20	
		Batch	Column	Batch	Column	Batch	Column	Batch	Column
API/incinerator ash									
	Acetate	11.3	8.4	10.7	8.1	10.5	8.0	10.1	7.7
	Carbonic acid	12.1	11.4	11.5	10.6	11.0	9.4	10.7	7.2
	Water	11.9	11.8	11.5	11.7	11.3	11.6	11.2	11.5
	ORNL/MWL	11.1	11.7	11.6	11.4	10.0	10.5	9.3	8.5
Dichloroethylene Still Bottoms									
	Acetate	4.79	4.80	4.83	4.90	4.87	4.90	4.89	4.90
	Carbonic acid	3.47	3.35	3.86	3.85	4.13	3.95	4.13	3.90
	Water	3.41	3.60	3.62	5.90	3.80	4.60	4.90	4.95
	ORNL/MWL	4.78	5.10	4.99	5.30	5.07	5.30	5.17	5.20
Paint sludge									
	Acetate	5.06	4.99	5.05	4.93	4.97	4.92	5.01	9.92
	Carbonic acid	5.31	5.43	5.35	5.16	5.31	5.03	5.15	4.76
	Water	5.34	5.35	5.42	5.80	5.48	6.00	5.50	6.20
	ORNL/MWL	5.31	5.35	5.30	5.35	5.29	5.30	5.27	5.25
Electroplating waste									
	Acetate	7.75	6.25	7.22	5.60	6.72	5.30	6.05	5.15
	Carbonic acid	7.93	7.00	7.57	6.41	7.25	5.87	6.78	5.72
	Water	8.98	9.18†	9.03	9.13	9.05	9.07	9.04	9.04
	ORNL/MWL	7.72	8.44	7.32	7.24	6.79	6.87	6.33	6.65

*Average of two replicates except where designated.

†Single analysis.

TABLE 46. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE MLC INORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

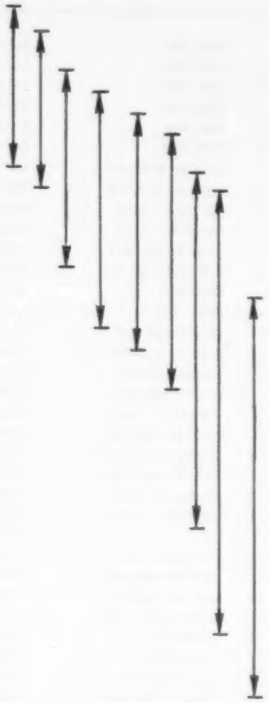
Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†
				Average	Minimum	Maximum		
1	ORNL/MWL	Column	10.0	50.2	10.8	97.7	60.2	
2	ORNL/MWL	Column	5.0	52.1	0.2	168.1	78.7	
3	Sodium acetate	Batch	10.0	53.6	15.5	90.5	46.0	
4	ORNL/MWL	Batch	5.0	54.2	2.2	100.0	55.8	
5	ORNL/MWL	Batch	10.0	55.7	3.0	100.0	48.0	
6	ORNL/MWL	Column	20.0	55.8	8.2	98.9	55.9	
7	ORNL/MWL	Batch	20.0	58.9	7.4	100.0	51.3	
8	Carbonic acid	Column	2.5	59.0	11.3	89.4	39.9	
9	Sodium acetate	Batch	20.0	59.1	7.3	100.0	55.8	
10	Sodium acetate	Batch	5.0	60.1	12.7	131.4	45.2	
11	Carbonic acid	Batch	5.0	60.6	4.5	97.5	52.0	
12	ORNL/MWL	Batch	2.5	61.8	7.6	108.5	55.6	
13	Carbonic acid	Column	5.0	63.0	6.9	87.2	41.4	
14	Sodium acetate	Column	2.5	64.0	31.8	119.5	32.9	
15	Carbonic acid	Batch	2.5	64.6	12.5	106.4	46.1	
16	Sodium acetate	Column	5.0	67.2	30.0	106.0	27.7	
17	Distilled water	Batch	2.5	67.6	8.2	149.2	55.1	
18	Carbonic acid	Batch	10.0	68.7	19.8	93.9	33.3	
19	Distilled water	Column	2.5	69.3	29.0	99.9	39.0	
20	ORNL/MWL	Column	2.5	69.7	11.6	281.5	87.9	
21	EP	Batch	20.0	69.9	1.9	188.7	65.3	
22	Distilled water	Batch	5.0	70.7	11.9	106.0	42.3	
23	Carbonic acid	Batch	20.0	70.8	30.1	93.2	27.0	
24	Carbonic acid	Column	10.0	71.2	23.0	90.2	31.1	
25	Sodium acetate	Column	10.0	71.2	40.5	91.7	23.8	
26	Distilled water	Column	5.0	72.4	12.5	99.9	35.2	
27	Sodium acetate	Batch	2.5	73.7	0.0	230.6	69.5	
28	Sodium acetate	Column	20.0	74.5	43.5	95.0	23.4	
29	Carbonic acid	Column	20.0	77.1	37.8	94.3	24.8	
30	Distilled water	Batch	10.0	79.0	46.0	99.9	23.9	
31	Distilled water	Column	10.0	79.8	14.5	99.9	29.7	
32	Distilled water	Batch	20.0	83.7	10.1	99.9	26.7	
33	Distilled water	Column	20.0	84.5	11.5	99.9	27.3	
34	Bi-sequential	Batch	20.0	96.9	4.7	777.3	192.1	

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 47. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE MLC ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

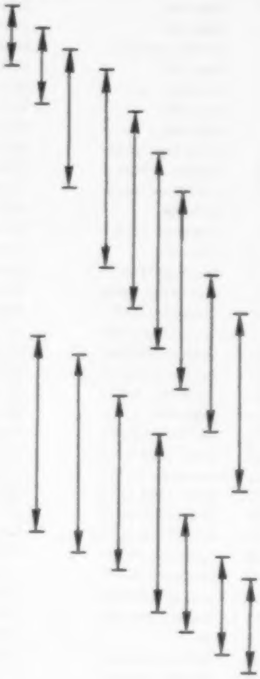
Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†
				Average	Minimum	Maximum		
1	ORNL/MWL	Column	5.0	68.2	9.5	99.0	43.4	
2	ORNL/MWL	Batch	2.5	69.0	29.7	94.7	31.4	
3	Sodium acetate	Batch	2.5	70.0	13.1	96.7	44.5	
4	ORNL/MWL	Column	2.5	73.8	20.1	100.0	39.4	
5	Carbonic acid	Batch	2.5	75.2	10.2	99.4	36.8	
6	ORNL/MWL	Batch	10.0	75.9	19.7	98.7	33.9	
7	ORNL/MWL	Column	10.0	77.3	39.1	99.5	28.1	
8	Distilled water	Batch	2.5	79.8	45.6	99.5	26.0	
9	Sodium acetate	Batch	5.0	80.7	45.6	96.0	22.2	
10	Distilled water	Column	2.5	83.6	48.8	99.9	24.7	
11	ORNL/MWL	Batch	5.0	83.8	53.2	97.9	17.5	
12	Carbonic acid	Batch	5.0	84.2	46.2	99.4	18.9	
13	ORNL/MWL	Column	20.0	84.8	52.5	99.7	17.7	
14	Bi-sequential	Batch	2.5	85.4	39.7	99.4	22.5	
15	Distilled water	Batch	5.0	86.5	64.8	97.4	13.2	
16	Carbonic acid	Batch	10.0	86.5	70.7	99.6	12.7	
17	Distilled water	Column	5.0	87.6	61.2	99.9	16.2	
18	Sodium acetate	Batch	20.0	88.6	72.8	97.2	8.8	
19	Sodium acetate	Batch	10.0	89.2	73.1	98.0	10.7	
20	Carbonic acid	Column	2.5	89.5	67.5	99.4	12.3	
21	ORNL/MWL	Batch	20.0	90.7	81.2	98.3	6.2	
22	Carbonic acid	Batch	20.0	90.9	80.0	99.7	8.3	
23	Distilled water	Column	10.0	91.6	79.4	99.9	9.7	
24	Carbonic acid	Column	5.0	91.8	79.8	99.4	8.2	
25	Distilled water	Batch	10.0	91.8	85.5	99.0	5.9	
26	Distilled water	Column	20.0	92.9	79.3	99.7	8.1	
27	Distilled water	Batch	20.0	93.2	83.6	100.0	6.8	
28	Carbonic acid	Column	10.0	93.4	86.3	99.6	6.2	
29	Carbonic acid	Column	20.0	94.2	85.5	99.8	6.2	
30	Sodium acetate	Column	20.0	94.6	74.3	99.4	9.1	
31	Sodium acetate	Column	10.0	95.5	82.2	99.4	6.0	
32	EP	Batch	20.0	95.7	89.3	100.0	3.9	
33	Sodium acetate	Column	5.0	97.4	94.3	99.4	1.8	
34	Sodium acetate	Column	2.5	98.2	92.7	99.4	2.1	



*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

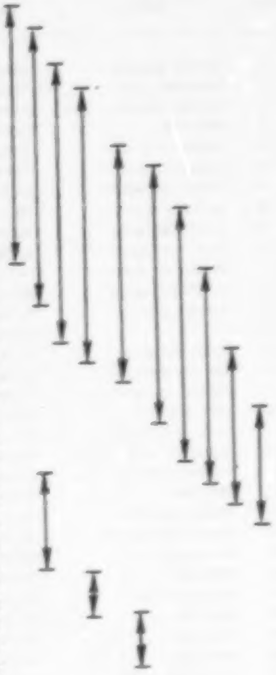
TABLE 48. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE MLC INORGANIC AND ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%) Average	Difference (%) Minimum	Difference (%) Maximum	Coefficient of variation (%)	Test for significance†
1	ORNL/MWL	Column	5.0	57.9	0.2	168.1	64.7	
2	ORNL/MWL	Column	10.0	59.9	10.8	99.5	50.2	
3	ORNL/MWL	Batch	10.0	63.0	3.0	100.0	44.0	
4	ORNL/MWL	Batch	2.5	64.4	7.6	108.5	46.8	
5	ORNL/MWL	Batch	5.0	64.8	2.2	100.0	45.0	
6	ORNL/MWL	Column	20.0	66.2	8.2	99.7	44.9	
7	Sodium acetate	Batch	10.0	66.4	15.5	98.0	40.2	
8	Sodium acetate	Batch	5.0	67.5	12.7	131.4	38.3	
9	Carbonic acid	Batch	2.5	68.4	10.2	106.4	42.3	
10	Carbonic acid	Batch	5.0	69.1	4.5	99.4	41.9	
11	Sodium acetate	Batch	20.0	69.7	7.3	100.0	43.2	
12	Carbonic acid	Column	2.5	70.0	11.3	99.4	35.3	
13	ORNL/MWL	Batch	20.0	70.4	7.4	100.0	40.8	
14	ORNL/MWL	Column	2.5	71.2	11.6	281.5	72.1	
15	Distilled water	Batch	2.5	71.9	8.2	149.2	44.9	
16	Sodium acetate	Batch	2.5	72.4	8.0	230.6	61.2	
17	Carbonic acid	Column	5.0	73.3	6.9	99.4	34.6	
18	Distilled water	Column	2.5	74.5	29.0	99.9	34.2	
19	Carbonic acid	Batch	10.0	75.1	19.8	99.6	28.0	
20	Sodium acetate	Column	2.5	76.3	31.8	119.5	31.0	
21	Distilled water	Batch	5.0	76.4	11.9	106.0	33.7	
22	Distilled water	Column	5.0	77.9	12.5	99.9	29.5	
23	Carbonic acid	Batch	20.0	78.1	39.1	99.7	23.8	
24	Sodium acetate	Column	5.0	78.1	38.0	106.0	26.8	
25	EP	Batch	20.0	79.2	1.9	188.7	48.4	
26	Carbonic acid	Column	10.0	79.2	23.0	99.6	26.3	
27	Sodium acetate	Column	10.0	80.0	40.5	99.4	22.8	
28	Sodium acetate	Column	20.0	81.7	43.5	99.4	21.6	
29	Distilled water	Batch	10.0	83.6	46.0	99.9	19.7	
30	Carbonic acid	Column	20.0	83.7	37.8	99.8	21.0	
31	Distilled water	Column	10.0	84.1	14.5	99.9	24.1	
32	Distilled water	Batch	20.0	87.1	10.1	100.0	21.4	
33	Distilled water	Column	20.0	87.5	11.5	99.9	21.9	
34	Bi-sequential	Batch	2.5	92.8	4.7	777.3	159.3	

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 49. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE AMCB INORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†
				Average	Minimum	Maximum		
1	Carbonic acid	Column	10.0	44.4	9.7	112.6	61.7	
2	Carbonic acid	Batch	20.0	45.6	0.7	101.9	61.9	
3	Carbonic acid	Column	20.0	51.6	12.6	80.2	41.8	
4	Carbonic acid	Column	5.0	55.3	9.1	214.1	93.1	
5	Sodium acetate	Column	20.0	61.4	22.8	151.0	54.1	
6	Distilled water	Column	10.0	62.5	13.7	119.2	52.6	
7	Carbonic acid	Batch	10.0	65.5	21.7	298.2	105.1	
8	Sodium acetate	Column	10.0	66.5	28.6	189.2	65.9	
9	ORNL/MWL	Batch	20.0	68.4	4.6	207.7	85.8	
10	Distilled water	Column	20.0	69.2	23.8	99.7	35.6	
11	Distilled water	Column	5.0	71.9	6.8	182.0	73.3	
12	Distilled water	Batch	20.0	75.7	19.2	113.3	32.9	
13	ORNL/MWL	Column	20.0	76.2	7.2	168.6	67.2	
14	Distilled water	Batch	10.0	80.9	21.9	241.3	76.6	
15	Sodium acetate	Column	5.0	83.1	18.2	203.0	70.6	
16	ORNL/MWL	Batch	10.0	87.5	24.6	308.3	83.4	
17	Sodium acetate	Batch	10.0	88.9	1.4	397.8	116.9	
18	Sodium acetate	Batch	20.0	90.7	21.6	468.9	118.5	
19	ORNL/MWL	Column	10.0	97.7	10.6	311.3	93.6	
20	EP	Batch	20.0	97.9	7.6	406.9	95.0	
21	Carbonic acid	Batch	5.0	101.7	3.9	511.5	127.2	
22	Carbonic acid	Column	2.5	103.3	9.1	375.4	87.2	
23	Distilled water	Batch	5.0	103.3	7.4	412.0	106.2	
24	Sodium acetate	Column	2.5	110.4	20.6	283.1	76.5	
25	Distilled water	Column	2.5	122.9	7.5	338.5	81.2	
26	Sodium acetate	Batch	5.0	132.2	6.8	781.8	152.4	
27	ORNL/MWL	Batch	5.0	136.2	14.2	405.8	88.8	
28	ORNL/MWL	Column	5.0	137.5	11.6	556.1	119.5	
29	Distilled water	Batch	2.5	171.9	2.1	611.1	93.7	
30	Carbonic acid	Batch	2.5	175.1	23.7	639.6	98.2	
31	ORNL/MWL	Batch	2.5	201.2	12.0	539.9	82.7	
32	ORNL/MWL	Column	2.5	214.5	14.9	810.2	109.2	
33	Sodium acetate	Batch	2.5	225.6	10.4	1037.8	121.2	
34	Bi-sequential	Batch	2.5	429.7	2.9	3625.5	206.6	

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

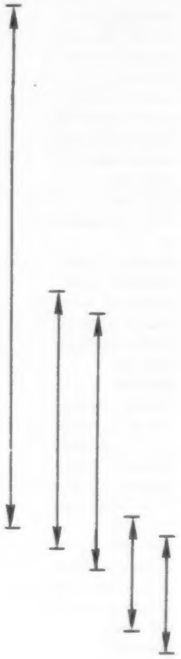
TABLE 50. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE AMCB ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%) Average	Difference (%) Minimum	Difference (%) Maximum	Coefficient of variation (%)	Test for significance†	
1	Sodium acetate	Batch	20.0	57.4	17.9	94.1	44.8	│	
2	Carbonic acid	Batch	10.0	62.4	23.3	99.0	41.1	│	
3	Sodium acetate	Batch	10.0	62.6	29.4	95.4	40.1	│	
4	ORNL/MWL	Column	20.0	67.2	10.7	92.6	43.4	│	
5	Carbonic acid	Column	2.5	67.4	15.5	99.0	42.6	│	
6	Carbonic acid	Column	5.0	68.2	9.4	99.0	45.2	│	
7	Distilled water	Batch	10.0	68.3	33.9	96.8	34.1	│	
8	Carbonic acid	Batch	20.0	71.2	34.2	99.0	34.1	│	
9	Distilled water	Batch	20.0	73.9	44.3	100.0	28.7	│	
10	Distilled water	Batch	5.0	74.4	22.4	132.1	45.0	│	
11	Distilled water	Column	10.0	75.4	41.9	99.7	32.9	│	
12	Distilled water	Column	20.0	75.5	53.0	99.5	25.2	│	
13	Carbonic acid	Column	10.0	76.6	49.8	99.0	26.4	│	
14	EP	Batch	20.0	76.8	14.9	100.0	34.6	│	
15	Carbonic acid	Column	20.0	81.2	50.7	99.0	21.4	│	
16	Sodium acetate	Column	10.0	81.2	33.7	99.0	26.9	│	
17	Sodium acetate	Column	20.0	82.5	41.6	99.0	24.7	│	
18	ORNL/MWL	Column	10.0	82.6	25.1	139.1	44.8	│	
19	Distilled water	Column	5.0	82.6	49.3	108.8	27.7	│	
20	Carbonic acid	Batch	5.0	85.5	26.5	171.2	54.4	│	
21	Sodium acetate	Column	5.0	91.2	77.0	99.0	7.6	│	
22	ORNL/MWL	Batch	10.0	96.6	28.9	321.4	89.9	│	
23	Sodium acetate	Column	2.5	100.3	90.4	130.3	11.4	│	
24	Distilled water	Column	2.5	107.7	51.8	251.9	54.6	│	
25	Bi-sequential	Batch	2.5	108.3	37.8	271.8	66.6	│	
26	ORNL/MWL	Column	5.0	112.0	25.2	370.5	91.3	│	
27	Carbonic acid	Batch	2.5	122.9	21.6	328.5	91.9	│	
28	ORNL/MWL	Batch	20.0	124.7	38.9	604.3	145.1	│	
29	ORNL/MWL	Column	2.5	170.7	42.4	668.9	116.7	│	│
30	ORNL/MWL	Batch	5.0	209.0	50.8	498.5	77.8	│	│
31	Distilled water	Batch	2.5	251.0	65.7	1546.9	193.7	│	│
32	ORNL/MWL	Batch	2.5	259.9	46.5	1096.1	135.3	│	│
33	Sodium acetate	Batch	5.0	323.1	36.6	2235.6	222.0		│
34	Sodium acetate	Batch	2.5	515.6	52.9	3758.1	236.3		

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 51. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE AMCB INORGANIC AND ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

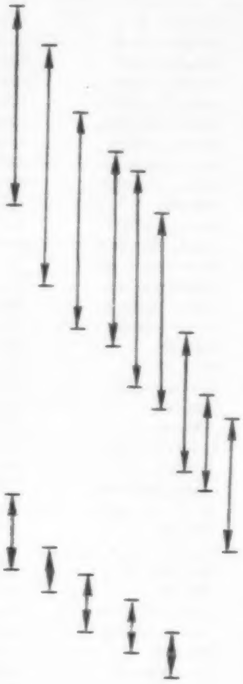
Rank	Media	Type	LS ratio*	Difference (%) Average	Difference (%) Minimum	Difference (%) Maximum	Coefficient of variation (%)	Test for significance†
1	Carbonic acid	Batch	20.0	54.8	0.7	101.9	53.2	
2	Carbonic acid	Column	10.0	56.0	9.7	112.6	52.2	
3	Carbonic acid	Column	5.0	59.9	9.1	214.1	74.8	
4	Carbonic acid	Column	20.0	62.3	12.6	99.0	39.4	
5	Carbonic acid	Batch	10.0	64.4	21.7	298.2	87.6	
6	Distilled water	Column	10.0	67.1	13.7	119.2	45.2	
7	Sodium acetate	Column	20.0	69.0	22.8	151.0	44.3	
8	Distilled water	Column	20.0	71.5	23.8	99.7	31.6	
9	Sodium acetate	Column	10.0	71.8	28.6	189.2	52.3	
10	ORNL/MWL	Column	20.0	73.0	7.2	168.6	60.4	
11	Distilled water	Batch	20.0	75.1	19.2	113.3	30.9	
12	Distilled water	Column	5.0	75.8	6.8	182.0	58.1	
13	Distilled water	Batch	10.0	76.4	21.9	241.3	67.0	
14	Sodium acetate	Batch	20.0	78.7	17.9	468.9	111.5	
15	Sodium acetate	Batch	10.0	79.4	1.4	397.8	106.2	
16	Sodium acetate	Column	5.0	86.0	18.2	203.0	54.3	
17	ORNL/MWL	Batch	20.0	88.7	4.6	604.3	132.6	
18	EP	Batch	20.0	90.3	7.6	406.9	84.0	
19	Carbonic acid	Column	2.5	90.4	9.1	375.4	83.2	
20	ORNL/MWL	Batch	10.0	90.8	24.6	321.4	84.3	
21	ORNL/MWL	Column	10.0	92.3	10.6	311.3	82.0	
22	Distilled water	Batch	5.0	92.9	7.4	412.0	96.9	
23	Carbonic acid	Batch	5.0	95.9	3.9	511.5	110.7	
24	Sodium acetate	Column	2.5	106.7	20.6	283.1	63.0	
25	Distilled water	Column	2.5	117.4	7.5	338.5	73.4	
26	ORNL/MWL	Column	5.0	128.3	11.6	556.1	111.6	
27	Carbonic acid	Batch	2.5	156.3	21.6	639.6	97.8	
28	ORNL/MWL	Batch	5.0	162.4	14.2	498.5	81.8	
29	ORNL/MWL	Column	2.5	198.8	14.9	610.2	110.2	
30	Distilled water	Batch	2.5	200.4	2.1	1546.9	155.0	
31	Sodium acetate	Batch	5.0	200.9	6.8	2235.6	225.7	
32	ORNL/MWL	Batch	2.5	222.4	12.0	1096.1	109.6	
33	Bi-sequential	Batch	2.5	314.0	2.9	3625.5	229.5	
34	Sodium acetate	Batch	2.5	330.0	10.4	3758.1	227.1	

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 52. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE AMC20 INORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%) Average	Difference (%) Minimum	Difference (%) Maximum	Coefficient of variation (%)	Test for significance†
1	Carbonic acid	Column	20.0	53.7	10.7	213.1	90.9	
2	Distilled water	Column	20.0	62.3	12.7	124.6	56.7	
3	Sodium acetate	Column	20.0	68.5	23.6	150.3	61.8	
4	Carbonic acid	Batch	20.0	77.0	1.9	411.3	143.0	
5	Carbonic acid	Column	10.0	79.8	11.4	331.4	104.0	
6	Distilled water	Column	10.0	82.6	9.2	190.6	70.0	
7	Distilled water	Batch	20.0	92.9	2.7	440.1	106.2	
8	Sodium acetate	Column	10.0	100.7	20.9	290.9	86.8	
9	ORNL/MWL	Batch	20.0	106.8	12.0	567.2	128.6	
10	ORNL/MWL	Column	20.0	107.1	12.9	473.8	107.5	
11	Distilled water	Batch	10.0	139.3	0.9	764.1	131.9	
12	EP	Batch	20.0	139.5	1.1	405.5	86.7	
13	Carbonic acid	Column	5.0	140.1	7.1	537.4	103.5	
14	Carbonic acid	Batch	10.0	142.4	10.1	908.1	158.0	
15	Distilled water	Column	5.0	155.9	8.4	433.0	80.2	
16	Sodium acetate	Column	5.0	156.1	24.5	509.4	91.9	
17	Sodium acetate	Batch	20.0	173.7	14.8	1340.2	185.5	
18	ORNL/MWL	Batch	10.0	178.1	28.6	785.2	117.3	
19	ORNL/MWL	Column	10.0	178.3	10.8	941.3	130.4	
20	Sodium acetate	Batch	10.0	199.4	23.3	1160.2	142.5	
21	Sodium acetate	Column	2.5	232.2	31.9	764.1	96.8	
22	Distilled water	Batch	5.0	233.3	6.6	1196.2	125.4	
23	Carbonic acid	Column	2.5	243.7	7.2	864.7	99.9	
24	Carbonic acid	Batch	5.0	254.5	10.4	1448.2	143.0	
25	Distilled water	Column	2.5	278.8	6.5	918.9	98.5	
26	ORNL/MWL	Column	5.0	298.2	11.8	1561.0	131.1	
27	ORNL/MWL	Batch	5.0	298.4	44.2	996.6	105.7	
28	Sodium acetate	Batch	5.0	329.9	14.5	2132.4	159.4	
29	Distilled water	Batch	2.5	384.6	44.2	1700.3	114.1	
30	Carbonic acid	Batch	2.5	401.9	22.1	1772.3	118.5	
31	ORNL/MWL	Batch	2.5	434.1	5.8	1484.3	102.7	
32	ORNL/MWL	Column	2.5	456.2	13.0	2204.4	122.7	
33	Sodium acetate	Batch	2.5	508.0	8.2	2780.5	138.5	
34	Bi-sequential	Batch	2.5	652.1	14.6	2714.1	128.0	



*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different (P<0.05) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 53. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE AMC20 ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†			
				Average	Minimum	Maximum					
1	Sodium acetate	Batch	20.0	58.7	4.8	94.1	46.7	│			
2	Carbonic acid	Batch	20.0	61.0	20.9	98.8	48.3	│			
3	Carbonic acid	Column	10.0	61.0	9.5	98.8	56.5	│			
4	Distilled water	Batch	20.0	62.8	23.9	100.0	45.6	│			
5	Distilled water	Batch	10.0	65.2	24.0	96.2	36.1	│			
6	Carbonic acid	Column	20.0	67.8	37.5	98.8	40.5	│			
7	Carbonic acid	Batch	10.0	67.8	8.8	202.1	87.4	│			
8	EP	Batch	20.0	71.0	24.4	100.0	36.7	│			
9	Sodium acetate	Column	20.0	71.1	12.2	98.8	46.0	│			
10	Distilled water	Column	20.0	72.8	27.6	133.8	51.8	│			
11	Carbonic acid	Column	5.0	75.6	42.8	121.8	37.9	│			
12	Sodium acetate	Column	10.0	76.6	37.7	98.8	27.2	│			
13	Distilled water	Column	10.0	78.6	27.9	117.8	41.5	│			
14	Sodium acetate	Batch	10.0	82.9	5.9	185.9	59.0	│			
15	ORNL/MWL	Column	20.0	83.6	11.0	171.4	53.9	│			
16	Sodium acetate	Column	5.0	96.9	77.8	169.8	29.4	│	│		
17	Carbonic acid	Column	2.5	99.6	33.5	256.6	66.9	│	│		
18	Distilled water	Batch	5.0	112.9	56.4	177.4	41.8	│	│		
19	Distilled water	Column	5.0	114.2	22.0	325.2	81.2	│	│		
20	Sodium acetate	Column	2.5	119.9	88.5	323.0	63.5	│	│		
21	ORNL/MWL	Batch	20.0	133.0	9.7	762.8	178.8	│	│		
22	ORNL/MWL	Column	10.0	139.3	4.5	486.0	103.4	│	│		
23	Carbonic acid	Batch	5.0	142.5	36.0	490.3	112.7	│	│		
24	ORNL/MWL	Batch	10.0	156.7	35.5	416.3	88.9	│	│		
25	Bi-sequential	Batch	2.5	183.1	4.3	636.5	130.5	│	│		
26	Distilled water	Column	2.5	195.5	25.8	762.5	128.3	│	│	│	
27	ORNL/MWL	Column	5.0	224.4	15.1	1053.0	151.8	│	│	│	
28	Carbonic acid	Batch	2.5	236.3	20.0	886.3	132.0	│	│	│	
29	ORNL/MWL	Column	2.5	358.1	33.9	1784.4	164.2	│	│	│	│
30	ORNL/MWL	Batch	5.0	427.8	24.4	1366.7	115.3	│	│	│	│
31	Distilled water	Batch	2.5	483.5	55.9	3064.3	202.7		│	│	│
32	ORNL/MWL	Batch	2.5	543.8	17.7	2269.3	149.2			│	│
33	Sodium acetate	Batch	5.0	626.3	50.8	4501.8	233.1				│
34	Sodium acetate	Batch	2.5	1020.0	53.6	7542.3	241.2				

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different (P<0.05) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

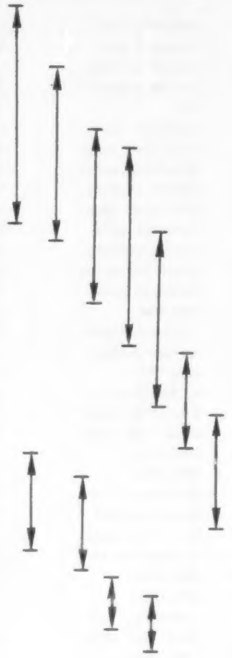
TABLE 54. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE AMC20 INORGANIC AND ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†				
				Average	Minimum	Maximum						
1	Carbonic acid	Column	20.0	58.8	10.7	213.1	72.0	│				
2	Distilled water	Column	20.0	66.1	12.7	133.8	54.1	│				
3	Sodium acetate	Column	20.0	69.5	12.2	150.3	55.4	│				
4	Carbonic acid	Batch	20.0	71.2	1.9	411.3	125.0	│				
5	Carbonic acid	Column	10.0	73.0	9.5	331.4	94.7	│				
6	Distilled water	Column	10.0	81.2	9.2	190.6	61.0	│				
7	Distilled water	Batch	20.0	82.1	2.7	440.1	98.8	│				
8	Sodium acetate	Column	10.0	92.0	20.9	290.9	77.3	│	│			
9	ORNL/MWL	Column	20.0	98.7	11.0	473.8	96.6	│	│			
10	Distilled water	Batch	10.0	112.6	0.9	764.1	133.5	│	│			
11	EP	Batch	20.0	114.8	1.1	405.5	89.2	│	│			
12	Carbonic acid	Batch	10.0	115.6	8.8	908.1	159.9	│	│	│		
13	ORNL/MWL	Batch	20.0	116.2	9.7	762.8	151.0	│	│	│		
14	Carbonic acid	Column	5.0	116.9	7.1	537.4	102.7	│	│	│		
15	Sodium acetate	Batch	20.0	132.3	4.8	1340.2	197.5	│	│	│		
16	Sodium acetate	Column	5.0	134.8	24.5	509.4	87.7	│	│	│		
17	Distilled water	Column	5.0	140.9	8.4	433.0	81.1	│	│	│		
18	Sodium acetate	Batch	10.0	157.5	5.9	1160.2	148.3	│	│	│		
19	ORNL/MWL	Column	10.0	164.2	4.5	941.3	123.4	│	│	│		
20	ORNL/MWL	Batch	10.0	170.4	28.6	785.2	108.0	│	│	│		
21	Distilled water	Batch	5.0	190.0	6.6	1196.2	126.4	│	│	│		
22	Sodium acetate	Column	2.5	191.8	31.9	764.1	99.7	│	│	│		
23	Carbonic acid	Column	2.5	191.8	7.2	864.7	108.7	│	│	│		
24	Carbonic acid	Batch	5.0	214.2	10.4	1448.2	143.5	│	│	│	│	
25	Distilled water	Column	2.5	248.8	6.5	918.9	106.1		│	│	│	
26	ORNL/MWL	Column	5.0	271.7	11.8	1561.0	135.5			│	│	
27	Carbonic acid	Batch	2.5	342.3	20.0	1772.3	124.2				│	│
28	ORNL/MWL	Batch	5.0	345.0	24.4	1366.7	111.2				│	│
29	Distilled water	Batch	2.5	420.2	44.2	3064.3	158.4					│
30	ORNL/MWL	Column	2.5	420.9	13.0	2204.4	133.0					│
31	Sodium acetate	Batch	5.0	436.6	14.5	4501.8	217.8					│
32	ORNL/MWL	Batch	2.5	473.6	5.8	2269.3	124.3					│
33	Bi-sequential	Batch	2.5	483.3	4.3	2714.1	147.4					
34	Sodium acetate	Batch	2.5	692.3	8.2	7542.3	223.3					

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 55. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE AMC40 INORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%) Average	Difference (%) Minimum	Difference (%) Maximum	Coefficient of variation (%)	Test for significance†
1	Distilled water	Column	20.0	99.6	2.7	209.2	50.1	
2	Carbonic acid	Column	20.0	111.4	11.1	442.8	98.8	
3	Sodium acetate	Column	20.0	115.8	4.3	389.2	94.1	
4	Distilled water	Batch	20.0	151.8	14.9	986.8	150.6	
5	Carbonic acid	Batch	20.0	166.1	2.4	928.9	148.0	
6	Distilled water	Column	10.0	174.3	12.2	460.3	69.0	
7	Carbonic acid	Column	10.0	188.9	11.8	647.8	99.9	
8	Sodium acetate	Column	10.0	198.6	34.2	686.7	95.1	
9	ORNL/MWL	Column	20.0	205.7	12.8	1054.6	123.7	
10	EP	Batch	20.0	223.6	34.7	769.5	92.6	
11	ORNL/MWL	Batch	20.0	230.2	12.2	1155.5	129.2	
12	Distilled water	Batch	10.0	279.3	34.7	1638.9	138.8	
13	Carbonic acid	Column	5.0	311.1	7.5	1004.9	101.3	
14	Carbonic acid	Batch	10.0	312.9	26.4	1928.8	153.0	
15	Sodium acetate	Column	5.0	313.2	30.3	1126.3	100.0	
16	Sodium acetate	Batch	20.0	324.2	13.1	2798.3	210.8	
17	Distilled water	Column	5.0	327.0	8.8	972.5	88.3	
18	ORNL/MWL	Column	10.0	360.4	10.8	1995.6	134.3	
19	ORNL/MWL	Batch	10.0	373.0	26.7	1565.7	123.1	
20	Sodium acetate	Batch	10.0	385.6	27.4	2436.0	152.1	
21	Sodium acetate	Column	2.5	469.4	32.2	1638.9	103.6	
22	Distilled water	Batch	5.0	479.2	11.9	2508.4	128.8	
23	Carbonic acid	Column	2.5	516.2	7.6	1572.1	100.6	
24	Carbonic acid	Batch	5.0	542.2	15.5	3015.7	140.3	
25	Distilled water	Column	2.5	576.2	6.9	1950.5	104.0	
26	ORNL/MWL	Column	5.0	591.3	11.8	3242.5	135.0	
27	ORNL/MWL	Batch	5.0	613.9	44.4	2073.7	113.1	
28	Sodium acetate	Batch	5.0	655.7	9.2	4392.4	164.5	
29	Distilled water	Batch	2.5	775.8	44.4	3522.9	119.1	
30	Carbonic acid	Batch	2.5	828.0	22.4	3667.8	120.2	
31	ORNL/MWL	Batch	2.5	882.5	5.6	3088.1	109.4	
32	ORNL/MWL	Column	2.5	899.1	13.4	4537.3	126.5	
33	Sodium acetate	Batch	2.5	1024.9	8.9	5696.6	139.3	
34	Bi-sequential	Batch	2.5	1059.4	11.5	4899.6	123.9	

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different (P<0.05) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

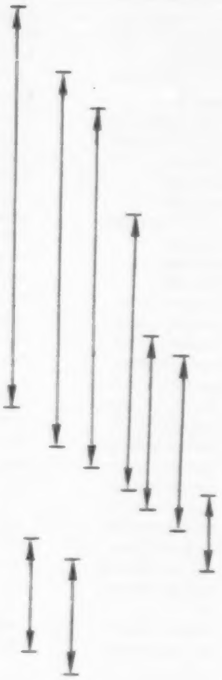
TABLE 56. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE AMC40 ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†			
				Average	Minimum	Maximum					
1	Carbonic acid	Column	20.0	55.9	4.9	98.7	69.3	│			
2	Carbonic acid	Batch	20.0	66.1	7.8	189.2	90.6	│			
3	Sodium acetate	Column	20.0	67.6	15.7	98.7	48.0	│			
4	Sodium acetate	Column	10.0	71.5	20.1	98.7	40.7	│			
5	EP	Batch	20.0	75.3	3.3	150.2	55.9	│			
6	Carbonic acid	Column	10.0	76.4	21.9	138.2	47.9	│			
7	Distilled water	Batch	20.0	81.7	26.2	168.6	55.9	│			
8	Distilled water	Column	20.0	85.9	6.5	198.9	67.9	│			
9	Sodium acetate	Batch	20.0	94.8	22.1	233.0	70.9	│			
10	Distilled water	Batch	10.0	95.8	24.4	218.7	76.2	│			
11	Carbonic acid	Batch	10.0	103.8	5.3	494.8	145.3	│			
12	Sodium acetate	Column	5.0	105.6	56.4	301.0	70.9	│			
13	Distilled water	Column	10.0	107.4	2.2	305.8	85.2	│			
14	Carbonic acid	Column	5.0	110.7	18.1	336.7	91.6	│			
15	ORNL/MWL	Column	20.0	121.4	14.1	434.2	104.2	│			
16	Sodium acetate	Batch	10.0	128.3	20.7	462.8	106.3	│			
17	Sodium acetate	Column	2.5	140.3	79.5	528.5	103.8	│			
18	ORNL/MWL	Batch	20.0	146.6	15.3	607.8	124.4	│	│		
19	Carbonic acid	Column	2.5	162.9	22.4	601.9	118.6	│	│		
20	Distilled water	Batch	5.0	171.2	43.8	446.1	88.9	│	│	│	
21	Distilled water	Column	5.0	186.9	4.3	737.0	132.3	│	│	│	
22	ORNL/MWL	Column	10.0	227.5	22.5	1053.4	150.6	│	│	│	
23	ORNL/MWL	Batch	10.0	231.9	51.4	789.5	113.4	│	│	│	
24	Carbonic acid	Batch	5.0	238.2	10.1	1061.8	145.3	│	│	│	
25	Bi-sequential	Batch	2.5	306.8	14.4	1202.4	147.8	│	│	│	
26	Distilled water	Column	2.5	344.0	5.0	1597.6	164.9	│	│	│	
27	Carbonic acid	Batch	2.5	404.8	12.4	1841.2	154.2	│	│	│	│
28	ORNL/MWL	Column	5.0	408.0	18.4	2169.2	179.8	│	│	│	│
29	ORNL/MWL	Column	2.5	655.4	14.1	3608.8	187.6	│	│	│	│
30	ORNL/MWL	Batch	5.0	739.2	25.7	2786.7	138.0		│	│	│
31	Distilled water	Batch	2.5	760.2	36.9	4574.5	194.6			│	│
32	ORNL/MWL	Batch	2.5	921.7	8.8	3420.3	151.2				│
33	Sodium acetate	Batch	5.0	961.9	29.6	6737.3	227.8				│
34	Sodium acetate	Batch	2.5	1576.9	33.6	11254.7	233.2				

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 57. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE AMC40 INORGANIC AND ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†
				Average	Minimum	Maximum		
1	Carbonic acid	Column	20.0	91.4	4.9	442.8	102.7	
2	Distilled water	Column	20.0	94.7	2.7	209.3	55.3	
3	Sodium acetate	Column	20.0	98.4	4.3	389.2	92.7	
4	Distilled water	Batch	20.0	126.6	14.9	986.9	146.8	
5	Carbonic acid	Batch	20.0	130.1	2.4	928.9	156.3	
6	Carbonic acid	Column	10.0	148.4	11.8	647.9	108.1	
7	Distilled water	Column	10.0	150.2	2.2	460.3	75.6	
8	Sodium acetate	Column	10.0	152.9	20.1	686.7	106.3	
9	EP	Batch	20.0	170.2	3.3	769.5	106.2	
10	ORNL/MWL	Column	20.0	175.4	12.8	1054.7	124.3	
11	ORNL/MWL	Batch	20.0	200.1	12.2	1155.5	130.3	
12	Distilled water	Batch	10.0	213.2	24.4	1639.0	151.0	
13	Carbonic acid	Batch	10.0	237.7	5.3	1928.8	169.0	
14	Sodium acetate	Column	5.0	238.5	30.3	1126.4	113.7	
15	Carbonic acid	Column	5.0	238.9	7.5	1004.9	114.7	
16	Sodium acetate	Batch	20.0	241.6	13.1	2798.3	228.9	
17	Distilled water	Column	5.0	276.6	4.3	972.6	100.5	
18	Sodium acetate	Batch	10.0	293.0	20.7	2436.0	166.2	
19	ORNL/MWL	Column	10.0	312.6	10.8	1995.6	139.4	
20	ORNL/MWL	Batch	10.0	322.2	26.7	1565.7	124.0	
21	Sodium acetate	Column	2.5	351.0	32.2	1639.0	121.1	
22	Distilled water	Batch	5.0	368.3	11.9	2508.5	140.7	
23	Carbonic acid	Column	2.5	389.1	7.6	1572.2	118.0	
24	Carbonic acid	Batch	5.0	432.8	10.1	3015.7	150.4	
25	Distilled water	Column	2.5	492.6	5.0	1950.6	119.1	
26	ORNL/MWL	Column	5.0	525.3	11.8	3242.6	145.7	
27	ORNL/MWL	Batch	5.0	659.0	25.7	2786.7	122.5	
28	Carbonic acid	Batch	2.5	675.6	12.4	3667.8	131.7	
29	Sodium acetate	Batch	5.0	765.9	9.2	6737.3	200.1	
30	Distilled water	Batch	2.5	770.2	36.9	4574.5	145.9	
31	Bi-sequential	Batch	2.5	788.5	11.5	4899.6	143.5	
32	ORNL/MWL	Column	2.5	811.3	13.4	4537.3	142.0	
33	ORNL/MWL	Batch	2.5	896.6	5.6	3420.3	123.7	
34	Sodium acetate	Batch	2.5	1223.6	8.9	11254.7	197.8	

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 58. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE IACB INORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†							
				Average	Minimum	Maximum									
1	Distilled water	Column	10.0	61.0	14.2	118.8	50.4	│							
2	Distilled water	Column	20.0	67.1	24.3	99.5	34.8	│							
3	Distilled water	Column	5.0	71.0	10.3	181.5	73.3	│	│						
4	Distilled water	Batch	20.0	73.3	17.4	113.3	34.0	│	│						
5	ORNL/MWL	Batch	20.0	76.2	2.4	224.0	96.4	│	│						
6	Distilled water	Batch	10.0	79.9	26.3	241.4	76.4	│	│						
7	ORNL/MWL	Batch	10.0	87.3	17.4	308.3	86.5	│	│						
8	Distilled water	Batch	5.0	102.6	6.8	412.0	107.8	│	│						
9	Distilled water	Column	2.5	121.9	7.5	347.9	84.7	│	│						
10	ORNL/MWL	Column	20.0	134.8	11.2	787.5	138.6	│	│						
11	ORNL/MWL	Batch	5.0	136.6	6.1	405.8	82.0	│	│						
12	ORNL/MWL	Column	5.0	142.8	11.5	556.1	114.8	│	│	│					
13	Distilled water	Batch	2.5	171.5	2.1	611.2	96.2	│	│	│	│				
14	ORNL/MWL	Batch	2.5	202.4	17.7	539.9	83.0	│	│	│	│				
15	ORNL/MWL	Column	10.0	209.8	10.5	1657.9	189.2	│	│	│	│				
16	ORNL/MWL	Column	2.5	214.8	15.4	810.3	110.4	│	│	│	│				
17	Carbonic acid	Batch	2.5	277.4	24.2	1700.0	150.5	│	│	│	│				
18	Carbonic acid	Column	20.0	426.0	13.1	6045.9	351.7	│	│	│	│	│			
19	Carbonic acid	Batch	5.0	432.9	3.9	5350.0	304.4	│	│	│	│	│			
20	Carbonic acid	Column	10.0	517.6	13.8	7601.8	364.9	│	│	│	│	│	│		
21	Carbonic acid	Column	5.0	638.9	9.6	9370.8	364.5	│	│	│	│	│	│	│	
22	Sodium acetate	Column	20.0	670.8	18.7	9596.4	354.9	│	│	│	│	│	│	│	
23	Carbonic acid	Column	2.5	679.7	9.7	9250.0	336.4		│	│	│	│	│	│	
24	Sodium acetate	Batch	2.5	739.7	10.9	8250.0	273.2			│	│	│	│	│	│
25	Carbonic acid	Batch	10.0	748.1	21.4	10950.0	363.7				│	│	│	│	│
26	Sodium acetate	Column	10.0	947.1	30.0	13914.0	365.2					│	│	│	│
27	Carbonic acid	Batch	20.0	1099.8	5.2	16900.0	383.0						│	│	│
28	Sodium acetate	Batch	20.0	1159.7	20.1	16900.0	362.0							│	│
29	Sodium acetate	Column	5.0	1179.9	17.3	17377.1	366.1							│	│
30	Sodium acetate	Column	2.5	1300.0	20.6	18900.0	361.1								│
31	Sodium acetate	Batch	5.0	1836.3	6.8	27400.0	371.3								
32	EP	Batch	20.0	1986.6	15.6	29900.0	374.7								
33	Sodium acetate	Batch	10.0	2368.8	9.2	36400.0	383.1								
34	Bi-sequential	Batch	2.5	4784.0	2.1	69900.0	363.4								

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 59. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE IACB ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†
				Average	Minimum	Maximum		
1	Sodium acetate	Batch	20.0	57.1	17.9	94.8	44.9	
2	Carbonic acid	Batch	10.0	62.0	23.3	99.0	41.4	
3	Sodium acetate	Batch	10.0	62.3	29.4	95.7	40.1	
4	Carbonic acid	Column	2.5	67.2	15.5	99.0	42.9	
5	Carbonic acid	Column	5.0	68.1	9.4	99.0	45.4	
6	Distilled water	Batch	10.0	68.4	33.9	96.8	34.3	
7	ORNL/MWL	Column	20.0	70.3	10.7	104.8	45.1	
8	Carbonic acid	Batch	20.0	71.3	32.5	99.0	34.6	
9	Distilled water	Batch	20.0	73.8	44.3	100.0	28.9	
10	Distilled water	Column	10.0	75.3	41.9	99.7	32.9	
11	Distilled water	Column	20.0	75.4	53.0	99.5	25.2	
12	Carbonic acid	Column	10.0	76.5	48.6	99.0	26.7	
13	Distilled water	Batch	5.0	76.6	22.4	151.8	49.7	
14	EP	Batch	20.0	76.7	14.9	100.0	34.7	
15	Carbonic acid	Column	20.0	81.1	49.4	99.0	21.7	
16	Sodium acetate	Column	10.0	81.2	33.7	99.0	26.9	
17	Sodium acetate	Column	20.0	82.4	41.6	99.0	24.7	
18	Distilled water	Column	5.0	82.5	49.3	108.8	27.8	
19	Carbonic acid	Batch	5.0	85.8	26.5	171.2	54.0	
20	ORNL/MWL	Column	10.0	86.8	25.1	162.6	49.9	
21	Sodium acetate	Column	5.0	91.1	77.0	99.0	7.6	
22	Sodium acetate	Column	2.5	100.3	90.2	130.3	11.5	
23	ORNL/MWL	Batch	10.0	103.4	28.9	384.0	103.7	
24	Distilled water	Column	2.5	107.7	51.8	251.9	54.7	
25	Bi-sequential	Batch	2.5	108.3	37.8	271.8	66.7	
26	ORNL/MWL	Column	5.0	116.2	25.2	370.5	88.0	
27	Carbonic acid	Batch	2.5	121.3	21.6	328.5	93.6	
28	ORNL/MWL	Batch	20.0	136.2	38.9	708.9	158.3	
29	ORNL/MWL	Column	2.5	174.9	42.4	668.9	114.0	
30	ORNL/MWL	Batch	5.0	213.5	50.8	498.5	77.1	
31	Distilled water	Batch	2.5	250.8	64.9	1546.9	193.9	
32	ORNL/MWL	Batch	2.5	262.1	46.5	1096.1	133.8	
33	Sodium acetate	Batch	5.0	322.7	36.6	2235.6	222.3	
34	Sodium acetate	Batch	2.5	515.4	52.9	3758.1	236.4	

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different ($P<0.05$) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 60. RANKING OF 34 LABORATORY EXTRACTION METHODS TO SIMULATE IACB INORGANIC AND ORGANIC TARGET CONCENTRATIONS FROM THE FIELD LYSIMETER TEST FACILITY

Rank	Media	Type	LS ratio*	Difference (%)			Coefficient of variation (%)	Test for significance†
				Average	Minimum	Maximum		
1	Distilled water	Column	10.0	66.2	14.2	118.8	44.0	
2	Distilled water	Column	20.0	70.1	24.3	99.6	31.1	
3	Distilled water	Batch	20.0	73.5	17.4	113.3	31.6	
4	Distilled water	Column	5.0	75.2	10.3	181.5	58.0	
5	Distilled water	Batch	10.0	75.7	26.3	241.4	66.6	
6	ORNL/PNL	Batch	10.0	93.1	17.4	384.0	92.8	
7	Distilled water	Batch	5.0	93.2	6.8	412.0	97.6	
8	ORNL/PNL	Batch	20.0	97.8	2.4	708.9	143.6	
9	ORNL/PNL	Column	20.0	111.6	10.7	787.5	136.3	
10	Distilled water	Column	2.5	116.8	7.5	347.9	76.0	
11	ORNL/PNL	Column	5.0	130.2	11.5	556.1	107.3	
12	ORNL/PNL	Batch	5.0	164.3	6.1	498.5	82.3	
13	ORNL/PNL	Column	10.0	165.5	10.5	1657.9	193.6	
14	Distilled water	Batch	2.5	200.0	2.1	1547.0	156.0	
15	ORNL/PNL	Column	2.5	200.4	15.4	810.3	110.2	
16	Carbonic acid	Batch	2.5	221.2	21.6	1700.0	156.0	
17	ORNL/PNL	Batch	2.5	223.9	17.7	1096.1	109.0	
18	Carbonic acid	Column	20.0	301.9	13.1	6045.9	396.4	
19	Carbonic acid	Batch	5.0	307.9	3.9	5350.0	342.8	
20	Carbonic acid	Column	10.0	358.8	13.8	7601.8	420.6	
21	Carbonic acid	Column	5.0	433.4	9.4	9370.8	429.7	
22	Sodium acetate	Column	20.0	459.0	18.7	9596.4	414.8	
23	Carbonic acid	Column	2.5	460.2	9.7	9250.0	399.1	
24	Carbonic acid	Batch	10.0	501.1	21.4	10950.0	434.4	
25	Sodium acetate	Column	10.0	635.4	30.0	13914.0	435.5	
26	Sodium acetate	Batch	2.5	650.0	10.9	8250.0	265.4	
27	Carbonic acid	Batch	20.0	729.6	5.2	16900.0	461.7	
28	Sodium acetate	Batch	20.0	762.8	17.9	16900.0	440.9	
29	Sodium acetate	Column	5.0	787.9	17.3	17377.1	438.7	
30	Sodium acetate	Column	2.5	868.1	20.6	18900.0	432.8	
31	Sodium acetate	Batch	5.0	1291.4	6.8	27400.0	422.6	
32	EP	Batch	20.0	1299.0	14.9	29900.0	458.8	
33	Sodium acetate	Batch	10.0	1538.5	9.2	36400.0	472.1	
34	Bi-sequential	Batch	2.5	3100.7	2.1	69900.0	449.4	

*LS ratio = liquid/solid ratio.

†Any two laboratory extraction procedures connected together by a vertical line are not statistically different (P<0.05) using Duncan's multiple range test, the EP and bi-sequential methods excluded because these methods were not replicated.

TABLE 61. TARGET CONCENTRATIONS*

		Target set				
Waste	Chemical	MLC	AMC8	AMC20	AMC40	IAC8
API/incinerator ash						
	Calcium	1188	787	771	774	792
	Chromium	63	22	11	6	21
	Molybdenum	2.2	0.60	0.30	0.09	0.66
	Potassium	562	164	76	40	164
	Sodium	1904	617	258	105	617
	Strontium	3.0	2.1	1.5	1.3	2.1
Dichloroethylene still bottoms						
	Dichloroethane	49	26	26	25	30
	Hexachlorobutadiene	2651	90	45	31	90
	Trichloroethane	83	53	43	38	53
	Trichloroethylene	97	26	21	26	22
Paint sludge						
	Barium	2.3	0.35	0.14	0.07	0.35
	Zinc	220	77	35	20	72
	Ethoxyethanol	4729	1055	430	219	1055
	Ethoxyethyl acetate	1892	405	165	84	405
	Toluene	39	17	9.9	6.9	17
	Xylenes	614	269	174	136	269
Electroplating waste						
	Barium	0.47	0.28	0.28	0.30	0.25
	Boron	148	51	22	11	51
	Manganese	7.4	1.3	0.79	1.5	0
	Nickel	147	107	93	98	80
	Potassium	125	30	39	41	29
	Sodium	7058	1209	472	209	1209
	Strontium	1.1	0.32	0.34	0.36	0.31
	Zinc	149	85	85	79	49

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*Concentrations in mg/L.